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Insoluble Coatings for Stirling Engine Heat Pipe Condenser Surfaces

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PROJECT SUMMARY PAGE

Project No. 91-1-10.01-6551B

Contract No: NAS3-26325 LERC

PROJECT TITLE:

INSOLUBLE COATINGS FOR STIRLING ENGINE HEAT PIPE

CONDENSER SURFACES

COMPANY:

Thermacore, Inc. 780 Eden Road Lancaster, PA 17601

SUMMARY:

This report describes the work done by Thermacore, Inc., Lancaster, Pennsylvania, for the Phase I, 1992 SBIR National Aeronautics and Space Administration Contract Number NAS3-26325, "Insoluble Coatings for Stirling Engine Heat Pipe Condenser Surfaces". The work was performed between January 1992 and July 1992.

Stirling heat engines are being developed for electrical power generation use on manned and unmanned earth orbital and planetary missions. Dish Stirling solar systems and nuclear reactor Stirling systems are two of the most promising applications of the Stirling engine electrical power generation technology. The sources of thermal energy used to drive the Stirling engine typically are non-uniform in temperature and heat flux. Liquid metal heat pipe receivers are used as thermal transformers and isothermalizers to deliver the thermal energy at a uniform high temperature to the heat input section of the Stirling engine. The use of a heat pipe receiver greatly enhances system efficiency and potential life span.

One issue that is raised during the design phase of heat pipe receivers is the potential solubility corrosion of the Stirling engine heat input section by the liquid metal working fluid. This Phase I effort initiated a program to evaluate and demonstrate coatings, applied to nickel based Stirling engine heater head materials, that are practically "insoluble" in sodium, potassium, and NaK. This program initiated a study of nickel aluminide as a coating and developed and demonstrated a heat pipe test vehicle that can be used to test candidate materials and coatings.

Nickel 200 and nickel aluminide coated Nickel 200 were tested for 1000 hours at 800°C at a condensation heat flux of 25 W/cm². Subsequent analyses of the samples showed no visible sign of solubility corrosion of either coated or uncoated samples. The analysis technique, photomicrographs at 200X, has a resolution of better than 2.5 μ m (.0001").

The results indicate that the heat pipe environment is not directly comparable to liquid metal pumped loop data, that nickel aluminide is still a leading candidate for solubility corrosion protection, and that longer duration tests are required to reach a definitive conclusion whether coatings are required at all. Should further testing be required, the test vehicle and analytical tools have been developed.

SUBMITTED: <u>Letur</u>

Principal Investigator Signature

DATE: 4-28-93

1.0 INTRODUCTION

Stirling heat engines are being developed for use on manned and unmanned earth orbital and planetary surface missions for electrical power generation. Liquid metal heat pipe receivers are currently being used as heat transformers or isothermalizers to deliver thermal energy at a uniformly high temperature to the heat acquisition section of Stirling engines that are being operated to generate electrical energy. The thermal energy can be supplied by solar concentrators, by pumped liquid metal loops in nuclear reactors, or by the combustion of solid, liquid, or gaseous fuels. Two current examples of the liquid metal heat pipe receiver/Stirling engine generator are the Dish Stirling System and the Nuclear Reactor/Stirling Engine System.

Dish Stirling systems are one of the most promising applications of the Stirling engine technology. Solar energy is concentrated by a parabolic reflector and is directed to the heat input section of the Stirling engine. The heat input section typically consists of a series of small diameter (5-8 mm) tubes that contain the engine working fluid, hydrogen or helium, as it shuttles between the expansion space and the cooler. It has been shown both analytically and by test, that the optimum engine efficiency results when the heat acquisition section is heated at a uniform temperature. The engine hot end typically operates at 550-800°C. Therefore, the optimum engine efficiency results when all of the heater head tubes are operating uniformly at 550-800°C. A liquid metal heat pipe receiver is typically utilized for this purpose. Figure 1 is a sketch of a typical heat pipe solar receiver for planetary use. A variation of this type of receiver can also be produced for use in space (zero gravity).

Nuclear Reactor/Stirling Engine Systems are also being developed for electrical power generation for use in space. In this application, the nuclear reactor is typically cooled with a liquid metal pumped loop. The pumped loop is thermally coupled to the Stirling engine by a heat pipe receiver. One example of this type of receiver is shown schematically in Figure 2. In this example, the heat acquisition section consists of solid metal fins with internal helium or hydrogen passageways. As mentioned previously, the optimum engine efficiency results when the heat input fins are heated at a uniform temperature of 550-800°C. Therefore, liquid metal heat pipes are used to transfer the heat from the reactor primary cooling loop at a uniform temperature to the heat input fins of the Stirling engine.

FIGURE 1. STIRLING ENGINE HEAT PIPE SOLAR RECEIVER

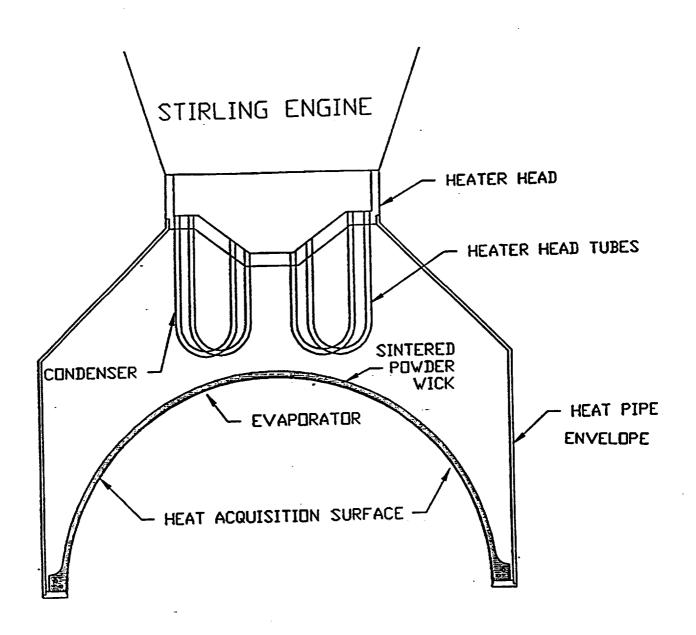
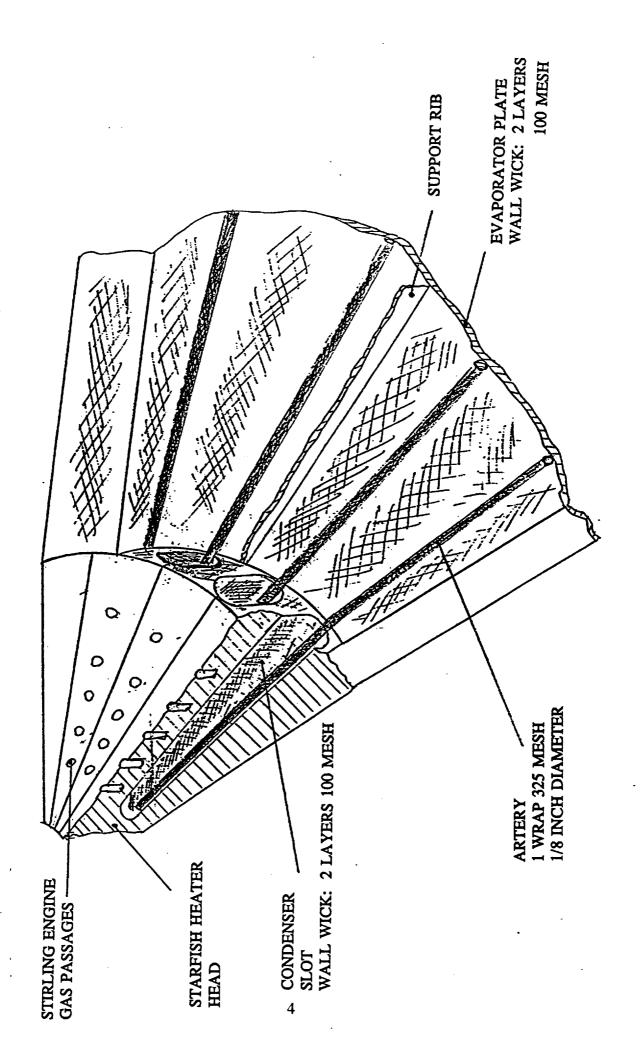


FIGURE 2. NUCLEAR REACTOR/STIRLING ENGINE HEAT PIPE RECEIVER

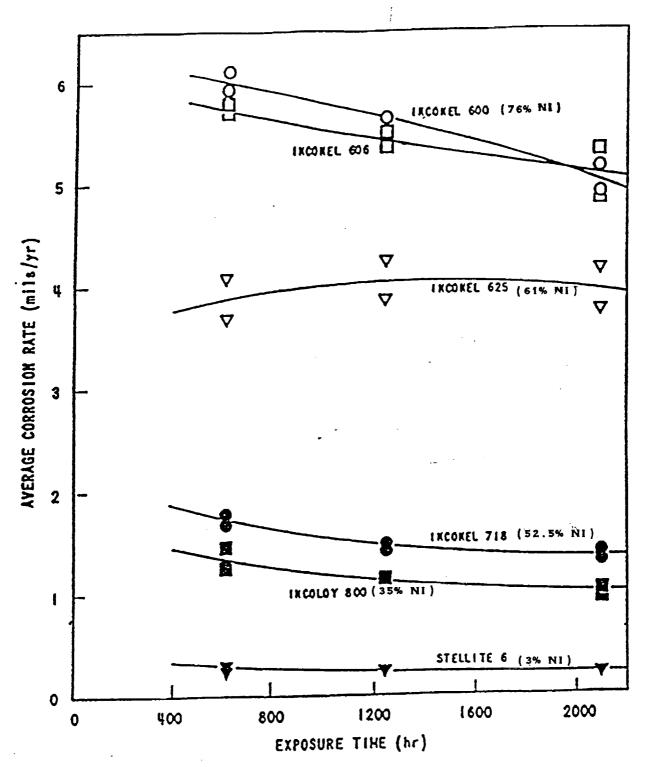


Heat pipes are sealed evacuated devices that transfer heat by evaporating and condensing a working fluid. Liquid metal heat pipes operating in the range of 550-800°C, typically utilize sodium as the working fluid. They are passive devices and do not require external power for operation. For the Stirling electrical power generation application, heat is input into the evaporator section which causes some of the working fluid to vaporize. The heat input can be non-uniform, at a higher or lower heat flux than the Stirling heat acquisition surface requires. The local vapor pressure in the evaporator section increases slightly over the pressure in the condenser section because of the heat input. This causes the vapor to flow to the condenser section where it condenses, releasing the latent heat of vaporization. This condensation process takes place on the exterior surface of the Stirling engine heat input tubes or fins. The condensation process delivers the thermal energy at a uniform temperature to the tubes or fins. The condensed fluid is then returned to the evaporator section by gravity and/or capillary pressure which is developed in the wick structure.

Materials of construction for high temperature heat pipes and the heat acquisition sections (heater head) of Stirling engines operating in the 550-800°C range are typically stainless steels and nickel based superalloys. At these operating temperatures, the constituents that make up these materials of construction are appreciably soluble in working fluids such as sodium, potassium, and NaK. Over the typical 7-10 years mission life span, essentially pure working fluid distillate will be condensing on the heat acquisition surfaces of the Stirling engine. This pure condensate will tend to leach the soluble constituents of the heater head material and transport them to the evaporator section of the heat pipe receiver. When the condensate is evaporated again, any soluble heater head materials are precipitated out and again nearly pure condensate will return to the heater head to leach more soluble material for transport back to the evaporator section. This continuous solubility cycle could corrode the heat input section of the Stirling engine. Solubility corrosion is a particularly important issue for the Stirling engine heat pipe receiver because of the relatively high condensation heat fluxes. Condensation heat fluxes of 20-25 W/cm² are common for this application. At 25 W/cm², a heater head for a 33% efficient 25 kWe Stirling engine will be washed with approximately 760,000 liters of sodium per year.

Solubility corrosion has the potential to be responsible for a number of premature failure mechanisms. Figure 3 is a plot of solubility corrosion rate versus time for a few nickel based superalloys subjected to flowing sodium at 720°C in a pumped loop. The corrosion rates

FIGURE 3. AVERAGE CORROSION RATE FOR NICKEL BASED ALLOYS
IN PUMPED LOOPS



SOURCE: Noble, J.E., Lehmann, G.A., Emigh, S.G., "Materials for a Stirling Engine Heater Head", Proceedings of the 25th IECEC, Paper No. 900272, August 12-17, 1990, Reno, Nevada - data from Westinghouse Hanford Company.

reported are 25-127 μ m/year (1-5 mils/year). Assuming a similar mechanism as that in a pumped loop, the wall thickness of a heater head tube or fin could be reduced by .25 - 1.27 mm (.010 - .050 inches) in ten years. In some cases this corrosion depth is greater than the starting wall thickness and in most cases the depth of corrosion is at least 50% of the starting wall thickness. Another potential failure mechanism is plugging of the heat pipe evaporator wick structure with the precipitated heater head material. Heat pipe wick structures typically have pore sizes of 1 x 10^{-5} m. Partial plugging can cause high liquid flow pressure drops that exceed the wick pumping capability which results in a dryout situation and potential melting of the receiver envelope.

A general trend for nickel based superalloys in pumped loop environments, which can be seen in Figure 3, is that higher nickel concentrations resulted in increased corrosion rates. This is only a trend and exceptions can be found. The other constituents, such as chromium, iron, molybdenum, columbium, aluminum, etc. are also soluble in sodium, potassium, and NaK. The literature suggests that iron is particularly susceptible to corrosion if the oxygen content of the liquid metal is high (>10ppm). Precipitation hardenable alloys are particularly susceptible to selective corrosion of the grain boundary materials. If this condition occurs the material may crack along the grain boundaries and cause failure of the pressure boundary.

Solubility of nickel based alloy constituents, preferential corrosion of grain boundaries, and the effects of excess oxygen in the working fluid are separate mechanisms that can cause corrosion of the heater head materials. There is also evidence that the synergistic effect of two or more of these mechanisms may further increase the rate of corrosion. These observations coupled with the fact that the heat pipe environment is in some ways more severe and in some ways less severe than pumped loop environments emphasizes the need for actual heat pipe tests to establish baseline solubility corrosion data for Stirling engine heat pipe receiver materials under simulated Stirling engine heat pipe conditions rather than relying on pumped loop data whose application to heat pipes is not established. Regardless of the desire for a one to one comparison, the pumped loop data already provide strong evidence that "insoluble" coatings for heater head surfaces will be required to meet the life and reliability requirements of Stirling engine system missions.

Metallurgical coatings, developed for wear and surface damage resistance for nickel based superalloys in liquid metal nuclear reactor systems, have been shown to greatly reduce the solubility corrosion (Johnson, R.N., "Tribological Coatings for Liquid Metal and Irradiation

Environments", Journal of Materials for Energy Systems, Vol. 8, No. 1, June 1986, p. 27-37). In particular, Johnson's testing of nickel aluminide diffusion coatings in sodium at 675° C has demonstrated the potential to reduce corrosion rates to approximately .76 μ m/year (.03 mils/year), 100 times less than the average uncoated nickel based alloys. This coating and others of similar characteristics have the potential to reduce corrosion rates to near negligible levels, essentially eliminate the effects of preferential grain boundary attack, and minimize the need for ultra pure working fluids. However, before these coatings will gain acceptance in the heat pipe industry for Stirling heat pipe receiver applications, a series of heat pipe tests must be run to demonstrate the performance under simulated Stirling engine system conditions.

The principal objective of this program is to develop an "insoluble" coating and coating application technique that can be used on Stirling engine heater head materials and for many other high temperature heat pipe applications where long, reliable life is a requirement. This is a Phase I SBIR (Small Business Innovative Research) program to address the issues described in this section. The specific objectives for the Phase I effort were the following:

- Evaluate alternative "insoluble" coating candidates and application techniques.
- Develop a test vehicle that can be used to experimentally evaluate candidate base materials and coatings under simulated Stirling engine conditions.
- Determine the baseline solubility corrosion rate of Nickel 200 and Nickel Aluminide coated Nickel 200 under simulated Stirling engine heat pipe receiver conditions.

2.0 TECHNICAL DESCRIPTION OF WORK PERFORMED

The Phase I program consisted of four technical tasks plus reporting. The first task was to establish a set of requirements for the Stirling engine environment. These include: working fluid, operating temperature, condensation heat flux, candidate heater head materials, and typical heater head geometries. These requirements were collectively determined based on current terrestrial and space Stirling programs. The second task was an evaluation of "insoluble" coating materials. A brief literature review was conducted and coating suppliers were interviewed for recommendations on solubility corrosion resistant materials. The third task was to prepare the sample tubes for the Phase I demonstration test vehicle. Three bare Nickel 200 tubes and three Nickel 200 tubes coated with nickel aluminide were prepared for test. The fourth task was to design, fabricate, and operate an electrically heated sodium heat pipe and associated gas gap calorimeters to simulate the Stirling engine environment per the requirements defined in Task 1. Following operation for 1000 hours at 800°C, the sample tubes were removed for analysis to determine the rate and/or depth of solubility corrosion.

The following sections detail the work completed during the Phase I effort.

2.1 TASK 1 - ESTABLISH REQUIREMENTS

Typical Stirling engine heat pipe receiver requirements were established by NASA LeRC and Thermacore personnel during the Phase I "kick-off" meeting. The requirements for a typical Stirling engine heat pipe receiver were selected based on a number of current Stirling engine heat pipe receiver programs. The requirements are generally more difficult for a space based application where maintenance is rarely available and long, reliable life is required for a successful mission. Therefore, the requirements selected were intentionally the most difficult to achieve and it is understood that successfully meeting the most difficult set of requirements means that all lesser sets of requirements are also feasible. Table 1 is a listing of the parameters and magnitudes selected for a typical Stirling engine heat pipe receiver.

TABLE 1. Stirling Engine Heat Pipe Receiver Requirements (Typical)

<u>Parameter</u>	<u>Magnitude</u>
Vapor Space Operating Temperature	750-800°C
Working Fluid	High Purity Sodium (≈ 10 ppm Oxygen)
Condensation Heat Flux	25 W/cm ²
Heater Head Geometries	Tubes or Fins
Heater Head Materials	Inco 718: UD720

The requirements for the Phase I test vehicle were also selected based on the following: the Phase I proposal, the typical requirements listed in Table 1, and the time and funding constraints of a Phase I program. The requirements for the Phase I test vehicle are listed in Table 2.

TABLE 2. Phase I Solubility Corrosion Test Vehicle Requirements

<u>Parameter</u>	Magnitude
Vapor Space Operating Temperature	800°C
Working Fluid	High Purity Sodium (Purchased, 10-20 ppm Oxygen)
Condensation Heat Flux	25 W/cm ²
Condensation Surface	25.4 mm (1.0") Diameter Tubes 101.6 mm (4.0") Long
Condenser Material #1	Nickel 200
Number of Samples, Material #1	3
Condenser Material #2	Nickel 200 with a 76.2 μ m (.003") 27% Aluminum Nickel Aluminide Coating
Number of Samples, Material #2	3

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2.2 TASK 2 - "INSOLUBLE" COATING MATERIAL EVALUATION

One of the goals of the Phase I effort was to determine candidate coating materials for the Stirling engine heat pipe receiver application. Candidate coating materials included: chromium carbide, select metal oxides, and nickel aluminide. Literature research indicated that chromium carbide was previously used in some high temperature sodium applications; however, chromium carbide was not chosen for the present application because of its unreliable bond strength with the substrate material. Metal oxides were dropped from consideration as coating materials as a result of insufficient available information. Because of its demonstrated bond strength in sodium cooled nuclear reactor applications, nickel aluminide was selected for Phase I application and testing.

Nickel aluminide coatings do have some disadvantages. The application or diffusion temperature is high, approximately 1000°C. This can be a problem for materials that require cold work for good mechanical properties. Also, mechanical properties gained from previous heat treatments may be lost during the high temperature diffusion process. However, many of the heater head designs are already brazed and/or vacuum fired at comparably high temperatures and the diffusion process should not affect the resulting mechanical properties for these designs.

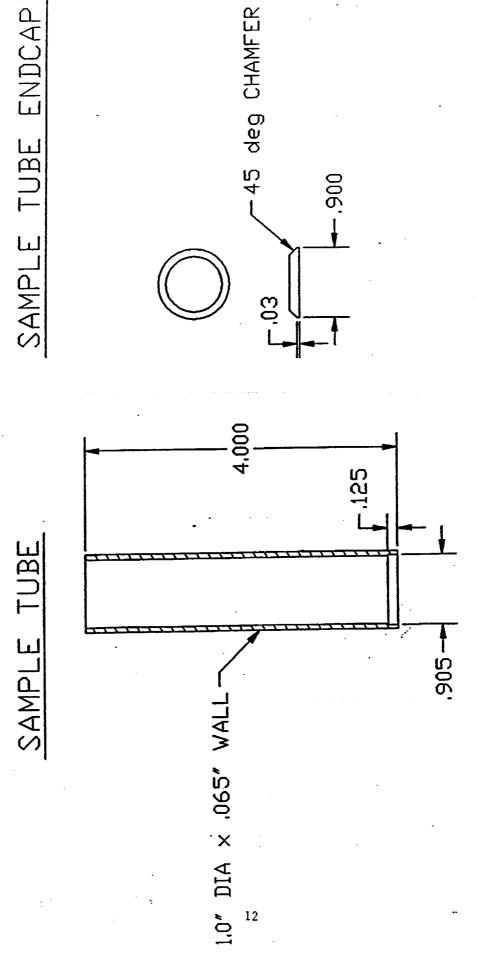
2.3 TASK 3 - SAMPLE TUBE PREPARATION

As stated in Section 2.1 Requirements, it was determined jointly by Thermacore and NASA LeRC personnel that a total of six sample tubes would be prepared for solubility corrosion testing. Three tubes would be Nickel 200 and three would be Nickel 200 with a nickel aluminide coating. Based on the condensation heat flux and the typical dimensions of a Stirling engine heater head, the sample tubes were chosen to be 25.4 mm (1.0") diameter x 1.65 mm (.065") wall x 101.6 mm (4") long. The sample tube and endcap drawings are shown in Figure 4. The endcaps were gas tungsten arc welded (GTAW) into the tubes.

Prior to the testing, the depth of corrosion for Nickel 200 and nickel aluminide coated Nickel 200 after 1000 hours at 800° C and 25 W/cm² condensation heat flux was projected to be on the order of 2.5-50.5 μ m (.0001"-.002"). These depths of corrosion were considered difficult to measure using standard micrometers and dial calipers. Therefore, alternative measuring techniques were evaluated.

FIGURE 4. SAMPLE TUBE AND END CAPS FABRICATION DRAWING

(Dimensions in inches)



MATERIAL! NICKEL 200

MATERIAL' NICKEL 200

Coordinate measuring machines are available with a resolution of 2.5 μ m (.0001"); however, there was a concern over the potential for thermal distortion of the sample tubes during the extended test period. Sectioning the samples after the test and taking photomicrographs was selected as the most accurate and economical method for this program.

Scaling from photomicrographs at high magnification, 50 to 400X, can be used to accurately determine the depth of corrosion. For example, at 200X magnification $2.5\mu m$ (.0001") of corrosion is .5 mm (.020") which is easily measured with an accurate flat scale or dial calipers. In order to make the measurements, a base measurement location must be preserved. One technique that can be used to preserve the base measurement location is to nickel plate stripes onto the sample tubes. The nickel plating stripes are applied axially, in the direction of gravity draining and protect the underlying surface from solubility corrosion. The nickel plating thickness was selected to be $76.2 \mu m$ (.003"). The nickel plating stripe must be thick enough such that the plating will not be completely dissolved during the 1000 hour test. Figure 5 illustrates the striped nickel plating concept.

One issue that remained was the concern that the nickel plating stripes may not adhere well to the nickel aluminide surface. Thermacore had on hand several INCO 718 plates that were nickel aluminide coated for a parallel effort. One of these plates was nickel plated and vacuum fired at 800°C for approximately 5 hours. The plate was subsequently potted, sectioned, and photomicrographed. The photomicrograph is shown in Figure 6. The nickel aluminide coating and the nickel plated stripe appear to be strongly adhered.

Chromalloy Research and Technology Division was contracted to coat several Nickel 200 sample tubes with nickel aluminide. Chromalloy typically uses one of two processes. The first, designated RT21, is a high temperature (1024°C) pack cementation diffusion coating process. This process is a one step process that is typically utilized for relatively thin coating thicknesses of 12.7-50.8 μ m (.0005" - .002"). The second process, designated RT573, is a two step process. The first step is a low temperature (746°C) pack cementation process. During this step, an essentially pure layer of aluminum is applied to the sample tube. The second step is a high temperature (1038°C) diffusion heat treat. This process is typically used when coating thicknesses greater than 50.8 μ m (.002") are required. Photomicrographs of each coating applied to a Nickel 200 sample tube are shown in Figures 7 and 8.

FIGURE 5. STRIPED NICKEL PLATING CONCEPT

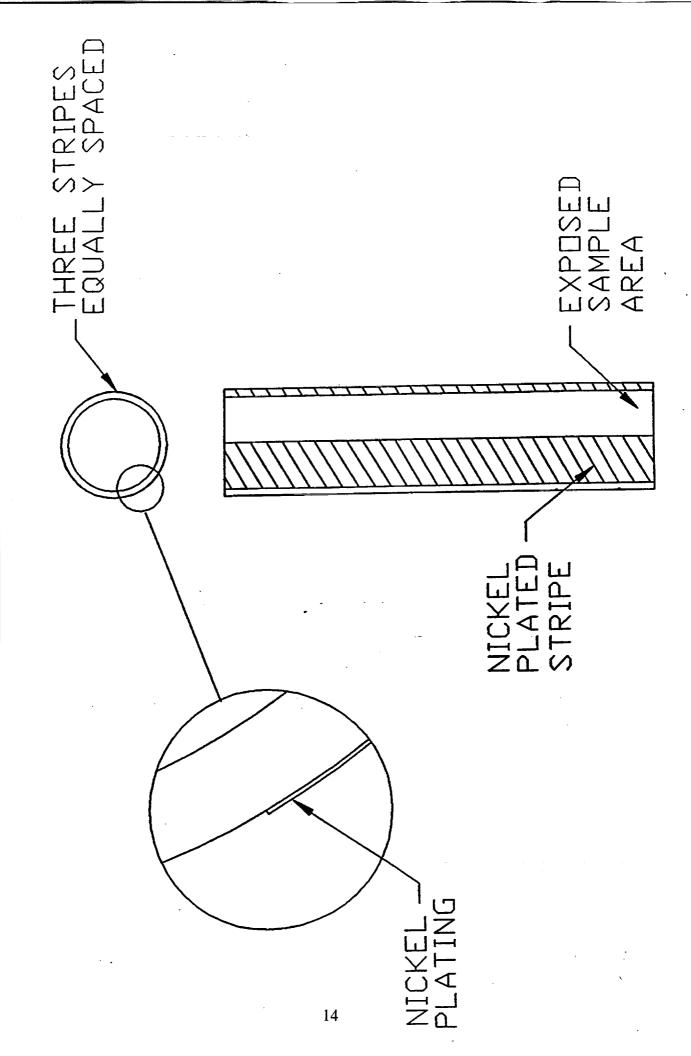


FIGURE 6. PHOTOMICROGRAPH NICKEL ALUMINIDE COATED INCO 718 PLATE WITH NICKEL PLATING STRIPES

THERMACORE, INC.

ALUMINIDE COATING ON INCO 718 PLATE

COATED BY CHROMALLOY RESEARCH & TECHNOLOGY DIVISION FOR MECHANICAL TECHNOLOGY, INC. 3 MILS

PLATED WITH 3 MILS, Ni-426 ELECTROLESS NICKEL VACUUM FIRED AT 820°C, 4-6 HOURS

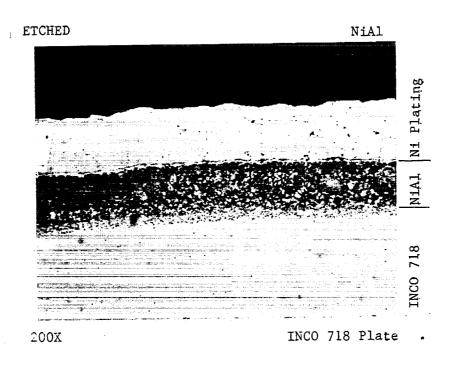
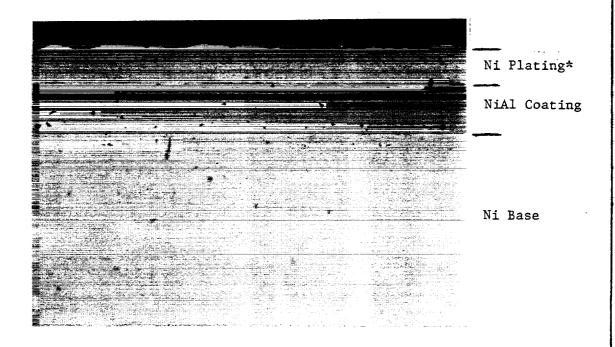


FIGURE 7. PHOTOMICROGRAPH OF NICKEL ALUMINIDE COATED NICKEL 200 TUBE, ONE STEP PROCESS, RT21

THERMACORE, INC

ALUMINIDE COATING ON NICKEL ALLOY TUBES EXTERNAL COAT ONLY

RT21 COATING, 1875 F 1.2 MILS, 27% AI



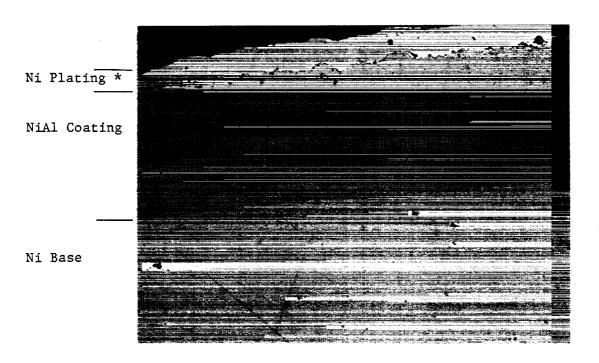
* Nickel Plating used to preserve NiAl surface for photomicrograph.

FIGURE 8. PHOTOMICROGRAPH OF NICKEL ALUMINIDE COATED NICKEL 200 TUBE, TWO STEP PROCESS, RT573

THERMACORE, INC

ALUMINIDE COATING ON NICKEL ALLOY TUBES EXTERNAL COAT ONLY

RT673 COATING, 1376 F 3.6 MILS, 27% AI



* Nickel plating used to preserve NiAl surface for photomicrograph.

Chuck Barner

Based on the long life requirement of the Stirling engine heater head and heat pipe receiver, the RT573 process was selected for this Phase I effort. A coating thickness of $76.2 \mu m$ (.003") was selected as a practical and conservative first test.

The sample tubes were dark green in color after coating. This was inconsistent with the INCO 718 samples that were used for plating adherence testing. Therefore, one of the sample tubes was immediately sectioned and photomicrographed. The photomicrographs, an example is shown in Figure 9, were identical to the earlier sample tubes and plates. The dark green film, perhaps indicative of chromium oxide, was not visible on the photomicrographs. The green coating on identically processed non-tested tubes was easily removed with a soft wire wheel. However, the sample tubes that were used in the 1000 hour heat pipe were tested in the as received dark green state.

Six sample tubes were selected for the test apparatus. Three bare Nickel 200 tubes and three nickel aluminide coated Nickel 200 tubes were prepared for testing. All six sample tubes were electroless plated with a low phosphorous (1-2%) nickel plating in the striped pattern shown in Figure 5. The sample tubes were subsequently vacuum fired at 800°C for two hours. The purpose of this furnace run was to verify nickel plating adherence. The sample tubes were then GTAW into a Nickel 200 tube sheet that was the top endcap for the test heat pipe. At this point the sample tubes were fully prepared and were ready for final installation into the heat pipe. Figure 10 is a photograph of the sample tubes welded into the top endcap.

2.4 TASK 4 - CORROSION TEST HEAT PIPE FABRICATION AND TEST

This section has been subdivided into four Sections 2.4.1 through 2.4.4. The first section, Section 2.4.1, details the corrosion heat pipe test vehicle design. The second section, Section 2.4.2, describes the fabrication and processing of the test vehicle. The third section, Section 2.4.3, describes the 1000 hour life test. And, the fourth section, Section 2.4.4, details the test vehicle dissection and sample tube analyses.

2.4.1 Corrosion Test Heat Pipe Design

According to the design requirements described in Section 2.1, a sodium heat pipe was required to deliver 25 W/cm² condensation heat flux to the sample tubes at 800°C for a minimum of 1000 hours. The heat pipe test vehicle that was selected for fabrication is a variation of a Thermacore heat pipe design that was developed for testing Stirling engines in a

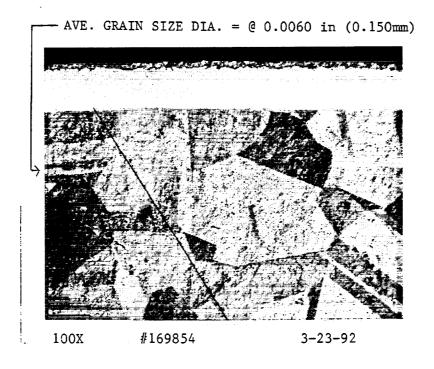
FIGURE 9. PHOTOMICROGRAPH OF "AS COATED" SAMPLE TUBE

THERMACORE, INC.

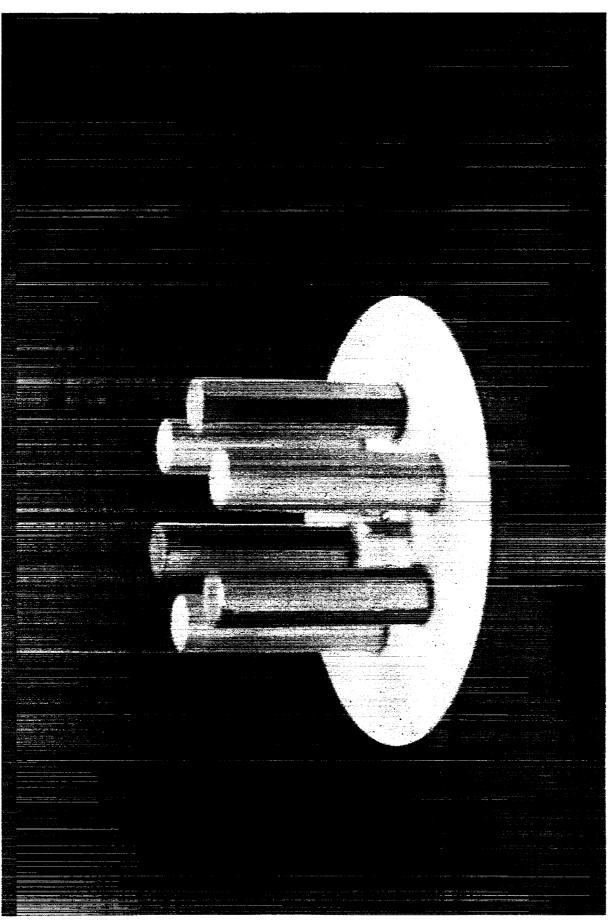
ALUMINIDE COATING ON NICKEL 200 TUBES

EXTERNAL COAT ONLY

COATED WITH CHROMALLOY RESEARCH & TECHNOLOGY DIVISION RT 673 COATING, 1375°F 3.6 MILS, 27% AI



SAMPLE #169854



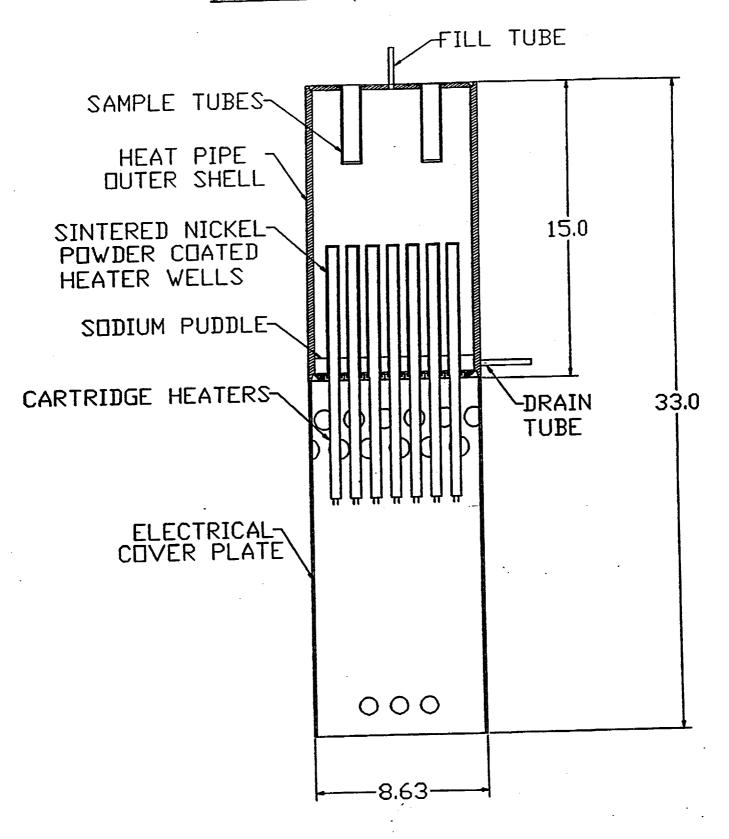
laboratory setting. A layout drawing of the selected design is shown in Figure 11. The evaporator section consists of 36, 1000 Watt cartridge heaters brazed into sintered nickel powder coated heater wells. The sodium fluid charge was selected to completely wet the sintered wick and to also cover the lower tube sheet to a depth of approximately 19 mm (.75"). The sodium working fluid is wicked up the length of the heater by capillary pressure generated in the sintered nickel wick structure. The sodium is evaporated from the heated surface and travels as a vapor to the condenser section consisting of the sample tubes welded into the heat pipe top endcap. Each of the sample tubes was fitted with a bayonet style gas gap calorimeter designed to extract the thermal power at the 25 W/cm² condensation heat flux.

The evaporator design is a variation of a number of heat pipes that Thermacore has supplied to Stirling engine manufacturers for laboratory testing of engines at full power conditions. The design of the nickel wick structure, 152.4 mm (6.0") long, 190.5 mm (.75") outer diameter, and 1.6 mm (.063") thickness was selected such that at full power (1000 Watts/heater), the safety factor for liquid pumping capability was 100% more than required. In other words, the wick structure was designed with a safety factor of 2 at the worst case operating conditions. In reality, the heaters are never operated at full power. In fact the heaters for this test vehicle were operated at only 40% of full power. Therefore, the effective wick design safety factor was approximately 5.

The reason for such a large safety factor is the uncertainty of cartridge heater life at 800°C. The maximum allowable heat flux for this type of heater, operating with an external surface temperature of 800°C in a zero clearance hole (brazed in design), is approximately 18 W/cm². To insure heater life in excess of one year, the heater manufacturer recommends that the internal heater element not be operated in excess of 870°C. Therefore, reduced heat fluxes and brazed in heaters were used to minimize the temperature difference between the internal heater element and the external surface of the heater. Based on Thermacore's experience with this type of heater, the manufacturer's recommendations appear to be conservative; however, because of the minimal increase in cost to obtain this level of reliability and the long time investment required to operate this type of test, the additional conservatism seems sensible.

In order to extract the thermal power and simulate the 25 W/cm² condensation heat flux, bayonet style gas gap calorimeters were designed. A gas gap calorimeter can be used to operate a heat pipe over a wide power range at a fixed operating temperature. The gas gap calorimeter

FIGURE 11. LAYOUT DRAWING OF THE SOLUBILITY CORROSION
TEST HEAT PIPE (Dimensions in inches)



consists of two parts: the heat pipe condenser tube (in this application the sample tube) and the water calorimeter. In this application, the water calorimeter is placed inside the condenser tube with a known gap between the two parts.

The operation of a gas gap calorimeter is best understood by tracing the flow of thermal energy from the inside of the heat pipe to the water in the water calorimeter. The sodium vapor generated in the evaporator section condenses on the outer diameter of the sample tubes releasing its latent heat of vaporization. The thermal energy is transferred through the wall of the sample tube by conduction. Next, the energy is transferred across the gas gap by radiation and by conduction through the gas. By controlling the fraction of argon, a low thermal conductivity gas, and the fraction of helium, a high thermal conductivity gas, in the gas gap, the resistance to conduction across the gap can be modulated. The heat is then transferred through the wall of the water calorimeter by conduction and transferred by convection to the water flowing through the water calorimeter. The temperature rise of the water flowing through the calorimeter and the water flowrate are used to calculate the power extracted and the condensation heat flux.

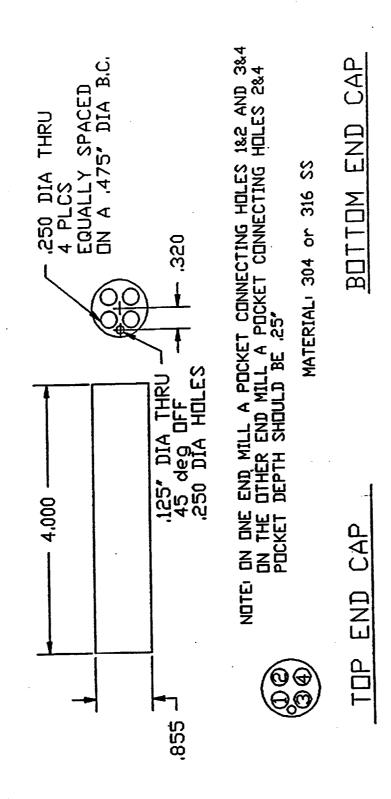
A summary of the gas gap calorimeter calculation results are shown in Table 3. The selected design and the "as fabricated" designs are highlighted in the table. Figure 12 is a drawing of the selected calorimeter design. The calorimeter water flows up and down through the 6.35 mm (.25") diameter holes and the gas is injected into the gas gap through the 3.17 mm (.125") diameter hole. A photograph of a fully assembled calorimeter and a sample tube are shown in Figure 13.

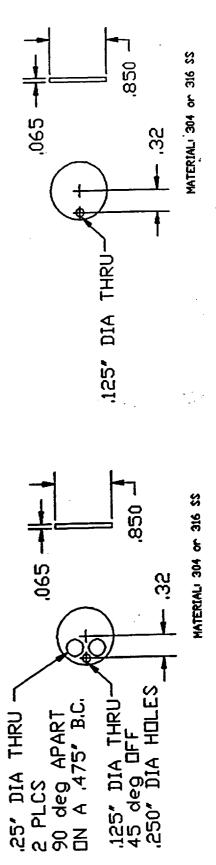
316 stainless steel was selected as the material of construction for the majority of the heat pipe envelope. The surfaces constructed of stainless steel were all well insulated such that the working fluid would not condense there to any significant degree. 316 stainless steel was selected because of its cost, availability, and durability in sodium heat pipes. The top endcap, the sample tubes, and the nickel wick structure were the only components made of another material. The nickel wick was selected for ease of fabrication and because of its proven performance and reliability. The top end cap was also fabricated from Nickel 200 to eliminate differential-thermal-expansion-induced stresses in the sample tubes. The Nickel 200 endcap also eliminates the potential of any of the constituents of stainless steel dissolving and affecting the solubility corrosion rate of the sample tubes.

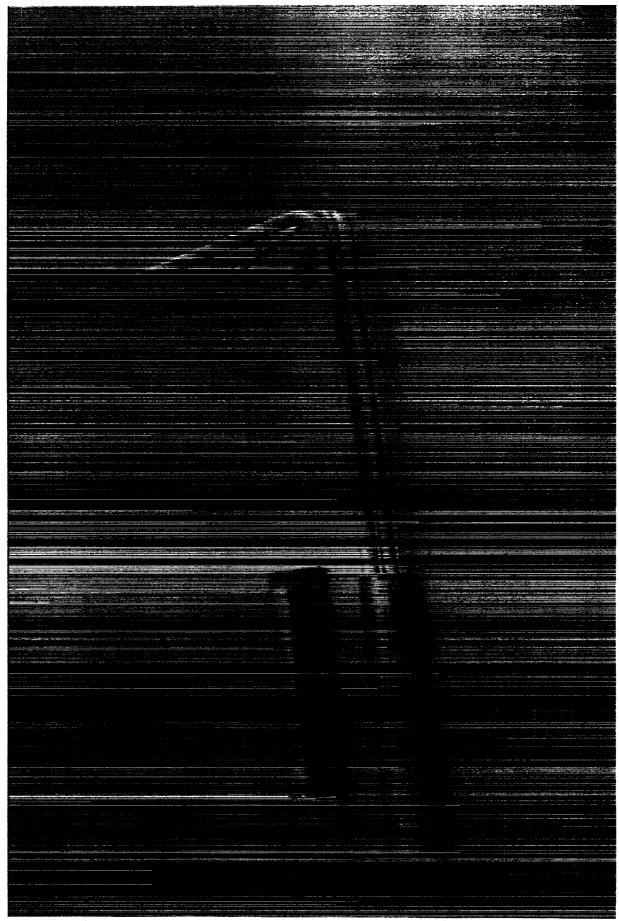
FIGURE 3. "INSOLUBLE" COATINGS FOR STIRLING ENGINE CONDENSER SURFACES GAS - GAP CALORIMETER CALCULATIONS

deg C INCHBS 600 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700 800 0.8700	INCHES									
DBSIGN		INCHES	INCHES		W/m - K	8	WATTS	WATTS	WATTS	W/CM ^ 2
DBRSIGN										
DBRSIGN	00 0.8775	0.8650	0.0063	ARGON	0.0287	0.092	541.45	159.85	701.30	11.56
DESIGN	00 0.8801	0.8650	0.0076	ARGON	0.0333	0.092	743.19	374.26	1117.45	18.42
DBSIGN						1		1		
DBSIGN	00 0.8775	0.8650	0.0063	HELIUM	0.2290	0.092	4327.80	139.83	4487.03	66.67
DESIGN	00 0.8801	0.8650	0.0076	HBLIUM	0.2700	0.092	6034.91	374.26	6409.17	105.67
DESIGN										
DHSIGN	00 0.8775	0.8600	0.0088	ARGON	0.0287	0.092	385.80	158.93	544.72	8.98
DBSIGN	00 0.8801	0.8600	0.0101	ARGON	0.0333	0.092	556.81	372.09	928.90	15.32
DESIGN										
DESIGN	00 0.8775	0.8600	0.0088	HELIUM	0.2290	0.092	3083.67	158.93	3242.60	53.46
DHSIGN	00 0.8801	0.8600	0.0101	HELIUM	0.2700	0.092	4521.44	372.09	4893.54	89.08
							Action 100 and			
	00 0.8775	0.8550	0.0113	ARGON	0.0287	0.092	299.26	158.01	457.27	7.54
	00 0.8801	0.8550	0.0126	ARGON	0.0333	0.092	444.65	369.93	814.58	13.43
	00 0.8775	0.8550	0.0113	HBLIUM	0.2290	0.092	2392.02	158.01	2550.02	42.04
	00 0.8801	0.8550	0.0126	HBLIUM	0.2700	0.092	3610.66	369.93	3980.59	65.63
		!								
	00 0.8775	0.8500	0.0138	ARGON	0.0287	0.092	244.17	157.08	401.26	6.62
	00 0.8801	0.8500	0.0151	ARGON	0.0333	0.092	369.73	367.77	737.50	12.16
		1				d	9	8	6	
008100	00 0.8775	0.8200	0.0138	HELIUM	0.2290	260.0	1931.09	\$0.751	77.9017	7.40
800	00,8801	0.8500	0.0151	HELIUM	0.2700	0.092	3002.33	367.77	3370.10	55.57
AS FABRICATED										
600 0.8670	70 0.8745	0.8560	0.0092	ARGON	0.0287	0.092	363.99	158.19	522.18	8.61
800 0.8670	170 0.8771	0.8560	0.0105	ARGON	0.0333	0.092	529.16	370.36	899.52	14.83
		6			-	6	7000	9	20.67.61	** C2
600 0.8670		0.8300	0.0092	HELIOM	0.2290	260.0	27.60%	¥1.0C1		6.00
800 0.8670	120 0.8771	0.8560	0.0103	HBLIUM	0.2700	0.092	4296.93	370.36	4667.29	76.95

CALORIMETER TUBE







Bar W.

2.4.2 Heat Pipe Fabrication and Processing

Fabrication drawings of the component parts were made and materials were ordered. The materials were sent out for machining. The first step in the fabrication process was to weld the endcaps into the heater wells. The next step was to nickel plate the heater wells with a low phosphorous, electroless nickel plating. This plating increases the wall wick bond strength between the heater well tube and the sintered nickel powder wick. Following plating, the nickel powder metal wick structures were sintered to the heater well tubes. The heater well tubes were then welded in the heater well tubesheet, which was also the bottom endcap for the heat pipe. The cartridge heaters were then inserted into the heater wells and furnace brazed in a hard vacuum. The sample tubes were also welded into the top endcap. Following the brazing operation, the entire heat pipe was then welded together. All of the welding was manual gas tungsten arc welding. The final step in the fabrication process was to vacuum fire the entire unit to degas the metal components and to remove adsorbed water and other solvents and oils. The unit was then prepared for charging and processing.

The heater leads were attached and wired in a balanced three phase delta arrangement. Commercially available high purity sodium (10-20 ppm oxygen) was pushed into the heat pipe test vehicle with high purity argon. The initial fluid charge was 675 cm³. The unit was operated for approximately four hours to temperatures up to 800°C. Non-condensible gases were observed as dark spots in low temperature areas in the upper portion of the heat pipe near the endcap. These gases were extracted, as required, through a vacuum line attached to the condenser endcap fill tube. After four hours, 450 cm³ of liquid sodium were drained out of the heat pipe. The purpose of the initial charge, operation, and subsequent discharge was to use the first sodium charge to react with and dissolve residual oxygen in the system. A second charge of 600 cm³ of liquid sodium was then added to the heat pipe. The heat pipe was operated for an additional two hours at 800°C before the fill tube was permanently sealed.

2.4.3 1000 hour Heat Pipe Test

Following the processing and sealing, the heat pipe was instrumented and setup for the 1000 hour life test. Instrumentation included the following:

- Three Type K vapor space thermocouples
- Six Type K water calorimeter inlet thermocouples

- Six Type K water calorimeter outlet thermocouples
- Six water calorimeter water flowmeters
- Six water calorimeter argon flowmeters
- Six water calorimeter helium flowmeters
- One electrical power Watt transducer

The water, argon, and helium flowrates were manually set, recorded and verified each day of the testing. The heat pipe temperature was controlled with a process temperature controller and a phase angle power controller. The control and instrumentation panel is shown schematically in Figure 14. Figure 15 is a photograph of the control and instrumentation panel and heat pipe.

The testing began on 5/12/92. Between 5/12/92 and 5/21/92, the heat pipe and gas gap calorimeters were only operated at ½ power for a total of 168 hours. The test was operated at ½ power until the water supply for the gas gap calorimeters could be made redundant and uniform in supply pressure. Since the heat pipe was operated at ½ power, for 168 hours this is equivalent to operating at full power for 84 hours. Between 5/21/92 and 6/29/92, the heat pipe was operated at full power for a total of 930 hours. The sum of the full power test hours and the equivalent ½ power test hours adds up to 1014 hours.

2.4.4 Heat Pipe Dissection and Sample Tube Analyses

Following the completion of the 1000 hour life test, a sample of the sodium was removed for future testing of oxygen content. The sample was taken in a 6.35 mm (.25") diameter x .89 mm (.035") wall tube that is 177.8 mm (7") long. After the sample was taken, the heat pipe was cut in half between the top of the cartridge heaters and the bottom of the sample tubes. The sodium was reacted in methanol. The sample tubes were removed from the tube sheet by grinding off the weld between the tube sheet and the sample tube.

The three Nickel 200 tubes looked identical and the 3 nickel aluminide coated tubes looked identical. Therefore only one of each sample was potted, sectioned and photomicrographed. Each of the sample tubes was sectioned at three locations; top (near nickel tube sheet), middle, and bottom (near sample tube endcap). At each of these three axial locations, three circumferential locations were photographed: between the nickel plating stripes, in the middle of a nickel plating stripe, and at the edge of a nickel plating stripe. Photographs were also taken in the unetched and etched states. The etchant which was used for the

FIGURE 14. SOLUBILITY TEST CONTROL AND INSTRUMENTATION PANEL LAYOUT

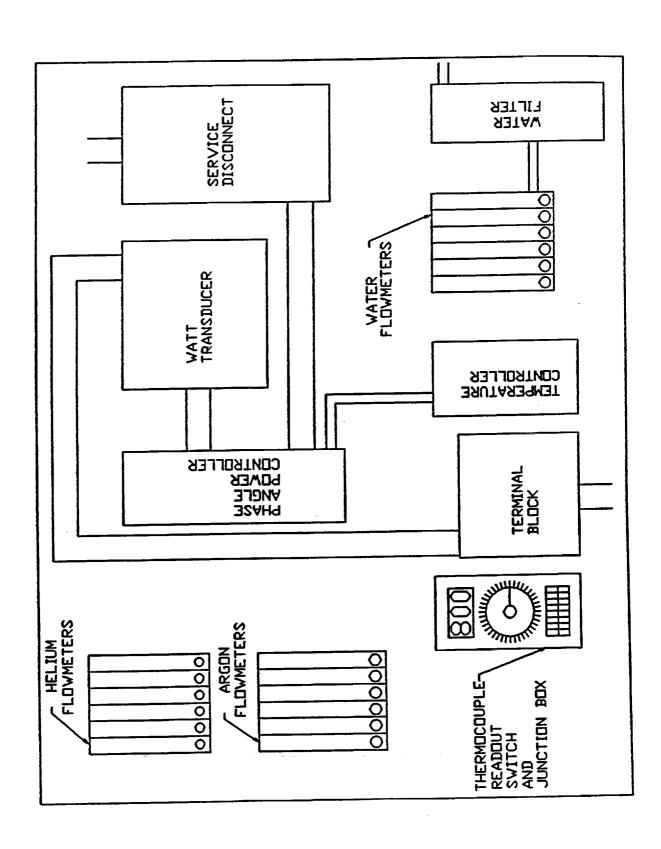
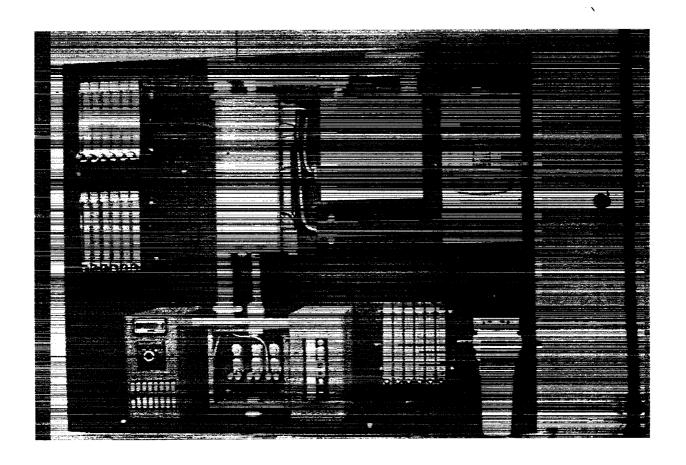


FIGURE 15. PHOTOGRAPH OF SOLUBILITY TEST HEAT PIPE AND CONTROL PANEL





photomicrographs was a three acid etch consisting of nitric acid, hydrochloric acid, and acetic acid. A small amount of cupric chloride was added to enhance the etchant's staining capability. Each of the above mentioned locations were photographed at three magnifications: 51, 100, and 200X. A complete set of photomicrographs is included as Appendix A. Figure 16 is a key of the axial and circumferential locations. Figure 19 presents the Appendix A photomicrograph labeling organization.

As discussed in Section 2.3, the photomicrograph technique has a resolution of 2.5 μ m (.0001") or better. For 200X photographs, .5 mm (.020") is equivalent to 2.5 μ m (.0001") of actual. An example of the nickel aluminide coated sample tube after the 1000 hour test is shown in Figure 17 along with a picture of a nickel aluminide coated sample tube before the 1000 hour test. The 1000 hour test appears to have had no affect on the nickel aluminide coated tube. The conclusion is that there is essentially no visible solubility corrosion, which quantitatively means less than 2.5 μ m (.0001") in 1000 hours. Figure 18 is a representative sample of the bare Nickel 200 sample tube. The Nickel 200 sample also shows no visible solubility corrosion, which quantitatively means less than 2.5 μ m (.0001") per 1000 hours. If solubility corrosion had occurred, the Figure 18 (top) photomicrograph surface feature would appear similar to the superimposed dashed line.

The striped nickel plating demonstrated strong adherence to both the nickel aluminide coated and the bare nickel tubes. Evidence of this adherence is shown in the Appendix A photomicrographs. The absence of large gaps between layers indicates the strong adherence. In the three nickel aluminide coated tubes, additional diffusion of the nickel aluminide coating into the nickel striping and nickel tube further contributed to bond adherence. This diffusion, however, produced an indeterminate initial reference line between the plating stripe and nickel aluminide coating. Because the solubility corrosion was nil, the reference line was not required for this case; however, alternative techniques may be required if substantial corrosion depths are observed.

All six sample tubes were measured on a coordinate measuring machine. However, without the starting measurement information, the after test measurements are not particularly useful for drawing quantitative conclusions. The measurements were consistent with the photomicrographs, although the accuracy was less. The experience did indicate a useful backup tool for future longer term tests.

FIGURE 16. KEY FOR AXIAL AND CIRCUMFERENTIAL PHOTOMICROGRAPH LOCATIONS

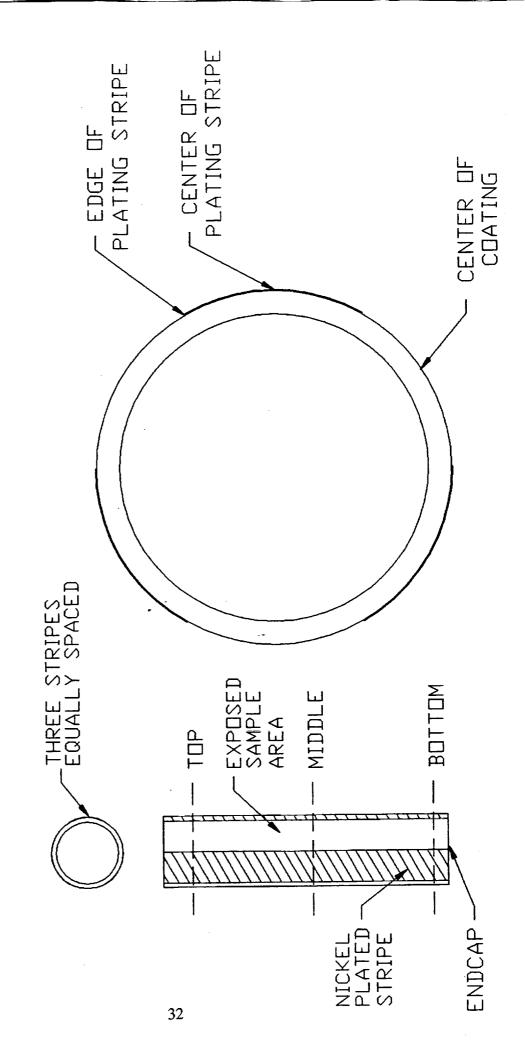
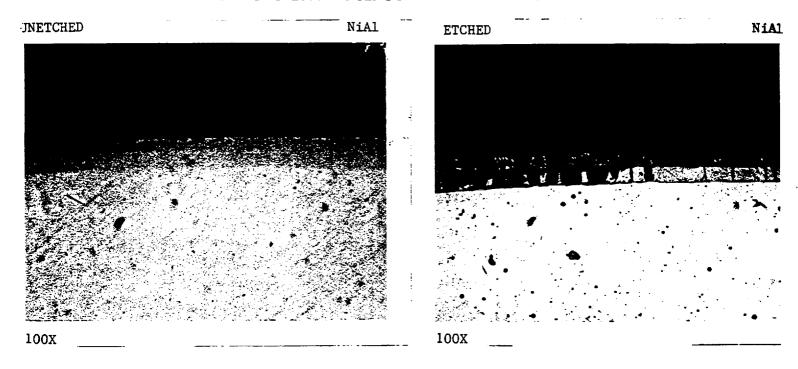


FIGURE 17. PHOTOMICROGRAPH OF NICKEL ALUMINIDE COATED NICKEL 200 SAMPLE TUBE BEFORE AND AFTER 1000 HOUR TEST

BEFORE 1000 HOUR CONDENSING SODIUM TEST



AFTER 1000 HOUR AT 800°C SUBJECTED TO 25 W/cm² CONDENSING HEAT FLUX WITH SODIUM

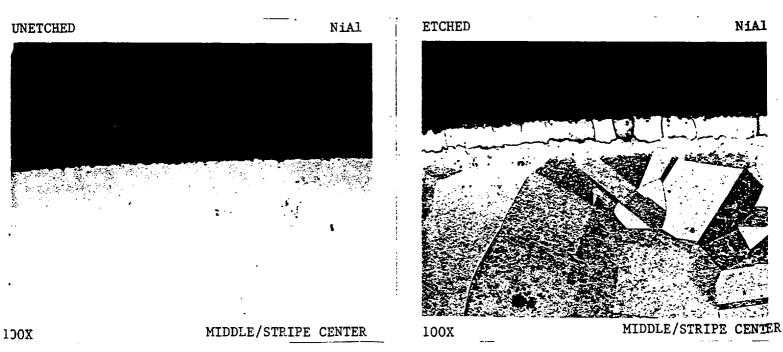
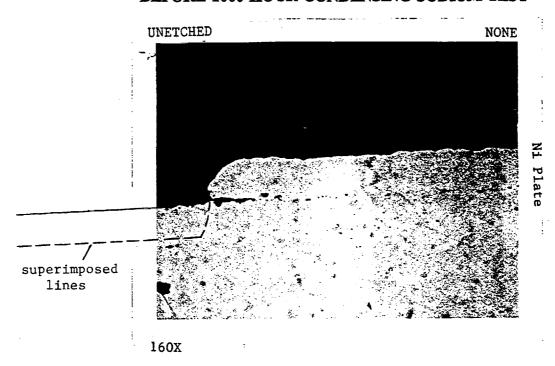
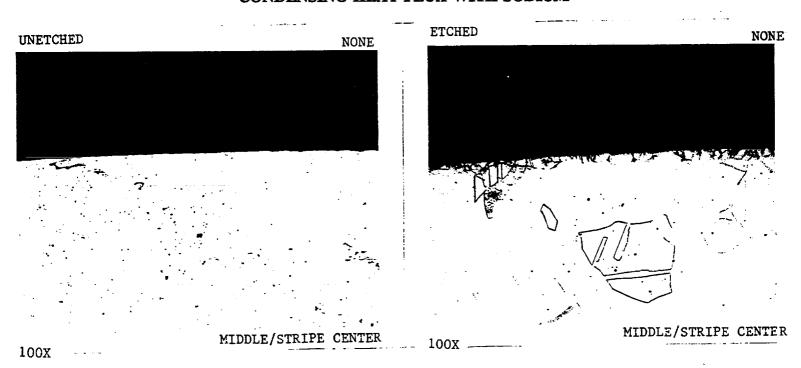


FIGURE 18. PHOTOMICROGRAPH OF NICKEL 200 SAMPLE TUBE BEFORE AND AFTER 1000 HOUR TEST

BEFORE 1000 HOUR CONDENSING SODIUM TEST



AFTER 1000 HOUR AT 800°C SUBJECTED TO A 25 W/cm² CONDENSING HEAT FLUX WITH SODIUM



3.0 CONCLUSIONS

Phase I of the program, "Insoluble Coatings for Stirling Engine Heat Pipe Condenser Surfaces", was very successful. The four contractual tasks were completed, including the preparation of six sample nickel aluminide coated and uncoated Nickel 200 tubes, the fabrication of an electrically heated sodium heat pipe and associated gas gap calorimeters to simulate the high condensation flux rate, and the performance of a 1000 hour life test and subsequent sample tube evaluation. The results, contrary to previous opinion and pumped loop experience, indicate the need for further study. The solubility corrosion rates determined from the 1000 hour life test were negligible in both coated and uncoated states, which is very encouraging for the Stirling engine application; however, the results indicate the need for additional, longer term tests.

The principle conclusions reached as a result of this effort are summarized below:

- 1. The test vehicle for subjecting samples to a simulated Stirling engine heater head environment was successfully demonstrated. The electrically heated sodium heat pipe was fabricated and operated for 1000 hours, delivering sodium vapor at 800°C. The unit operated at 14.4 kW_e and delivered 12 kW_{th} to the sample tubes. The gas gap calorimeters for heat removal and measurement demonstrated excellent control and feasibility. The heat pipe test vehicle also demonstrated the ease of sample tube installation and subsequent retrieval.
- 2. Photomicrographs of the nickel aluminide coated Nickel 200 tubes showed less than .00254mm (.0001") corrosion from the 25 W/cm² condensing sodium flux at 800°C for 1000 hours. The resolution of the photomicrographs is better than .00254 mm (.0001").
- 3. Photomicrographs of the bare Nickel 200 tubes showed less than .00254 mm (.0001") corrosion from the 25 W/cm² condensing sodium flux at 800°C for 1000 hours. The resolution of the photomicrographs is better than .00254 mm (.0001").

- 4. The rate of corrosion for the bare Nickel 200 is substantially less than that predicted by pumped loop data.
- 5. While plating stripes worked well for uncoated specimens, additional work is needed to perfect the technique for coated specimens. Physical measurements before and after test are also recommended for future tests.
- 6. Because the rates of solubility corrosion appear to be so small, longer test times on the order of 5,000 to 10,000 hours are recommended for future tests.

4.0 RECOMMENDATIONS

The results of this relatively short solubility corrosion life test have demonstrated the tools and time spans required to gain directly applicable test results. The test was important for two reasons. The nickel aluminide coating for solubility corrosion resistance is still a very attractive candidate for long life high temperature heat pipes; and, it has been shown under controlled conditions at high heat fluxes in a heat pipe that predictions of heat pipe corrosion based on pumped loop data are highly suspect. For these two reasons it is imperative to continue this effort. Without the information that this effort can gain, meaningful estimates of mission component reliability are indeterminate. The following is a brief list of recommendations pertaining to areas of the program that require additional research and development.

4.1 SURVEY OF SOLUBILITY CORROSION EXPERIENCE AND REQUIREMENTS

There are a great many researchers across the nation that have experience with solubility corrosion in liquid metal systems and who have requirements and goals for current and future high temperature heat pipe receiver applications. One of the first tasks for additional effort should include a letter or telephone survey that requests applicable input of past experience with liquid metal systems, input regarding any coatings work past or ongoing, and any additional requirements that are required to meet additional applications. The goal of this effort is to gain enough information such that the work effort can be tailored to solving real problems for real applications.

4.2 SOLUBILITY CORROSION TEST MATRIX

There are a number of materials of current and repeating interest with essentially no data on rates of solubility corrosion. Each time designs are proposed the same unanswered questions are posed. Therefore, in order to establish a limited data base, a test matrix of materials should be immediately placed on extended life test. As a baseline, comparable to a great deal of pumped loop data, Nickel 200 and 316 stainless steel should be tested in both the plain and nickel aluminide coated states. Haynes 230, a popular high strength material for terrestrial use, should also be tested as well as Inconel 718 and Udimet 720, which are proposed for the space

application. Each of these materials should also be tested in the coated and uncoated condition. These tests should be run in the test vehicle designed and demonstrated in Phase I. Additional materials may be added as required and indicated by the industry survey. These tests should be run a minimum of 1 year. Samples can also be fabricated with nickel or stainless steel screen wraps to determine the interaction between the wick structure and the wall material in a sodium environment.

4.3 COATING TECHNOLOGY FOR WICK STRUCTURES

If solubility corrosion is a substantial problem, the small diameter wires that make up screen wick structures with large surface areas are particularly susceptible. An effort should be undertaken to develop techniques for coating screen wick structures. Adapting the current nickel aluminide process for large surface area screens, weaving screen with nickel aluminide coated wires, and adding aluminum powder to the sodium working fluid which may react to form a nickel aluminide coating on all surfaces are all worth pursuing.

4.4 LONG LIFE SOLUBILITY RESISTANT HEAT PIPE

A demonstration heat pipe for the space Stirling power generator should be fabricated. The heat pipe should be as close to the proposed design as is feasible and should be operated indefinitely. This unit will pioneer the life expectancy path and will be a watch dog several years ahead of any units that are placed in service as a complete power generation system. Should a failure occur seven to ten years after beginning operation, the unit can be dissected and corrective action could then be determined for any in service units well ahead of a potentially catastrophic failure of an entire system. If the unit continues to operate successfully beyond ten years, missions could potentially be extended with confidence.

APPENDIX A

PHOTOMICROGRAPHS OF NICKEL 200

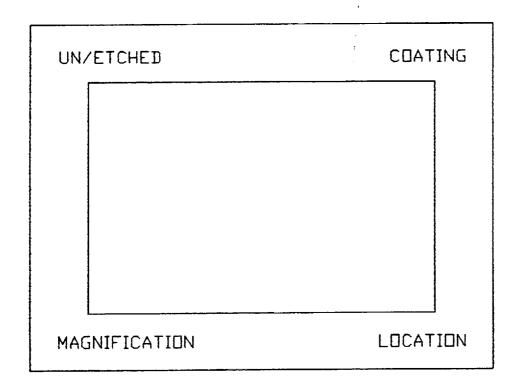
AND

NICKEL ALUMINIDE COATED NICKEL 200

SAMPLES AFTER 1000 HOURS AT 800°C

AND 25 W/cm² CONDENSING SODIUM HEAT FLUX

FIGURE 19. APPENDIX A PHOTOMICROGRAPH ORGANIZATION



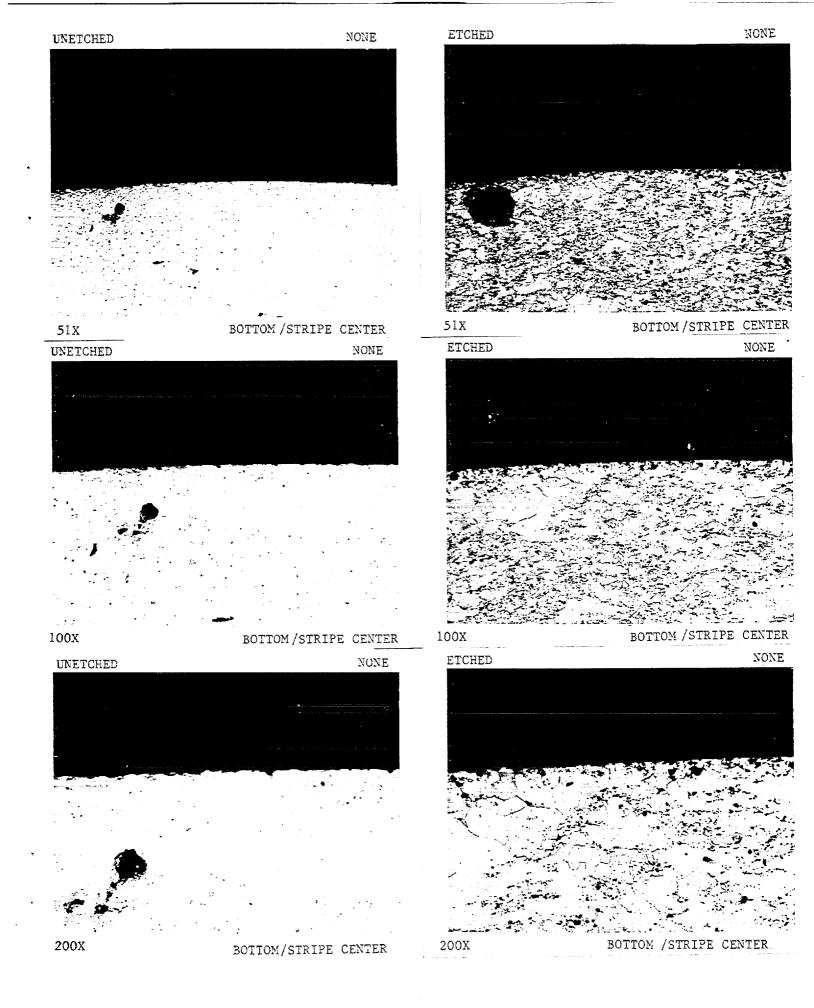
UN/ETCHED = UNETCHED OR ETCHED SAMPLE PHOTOMICROGRAPHS

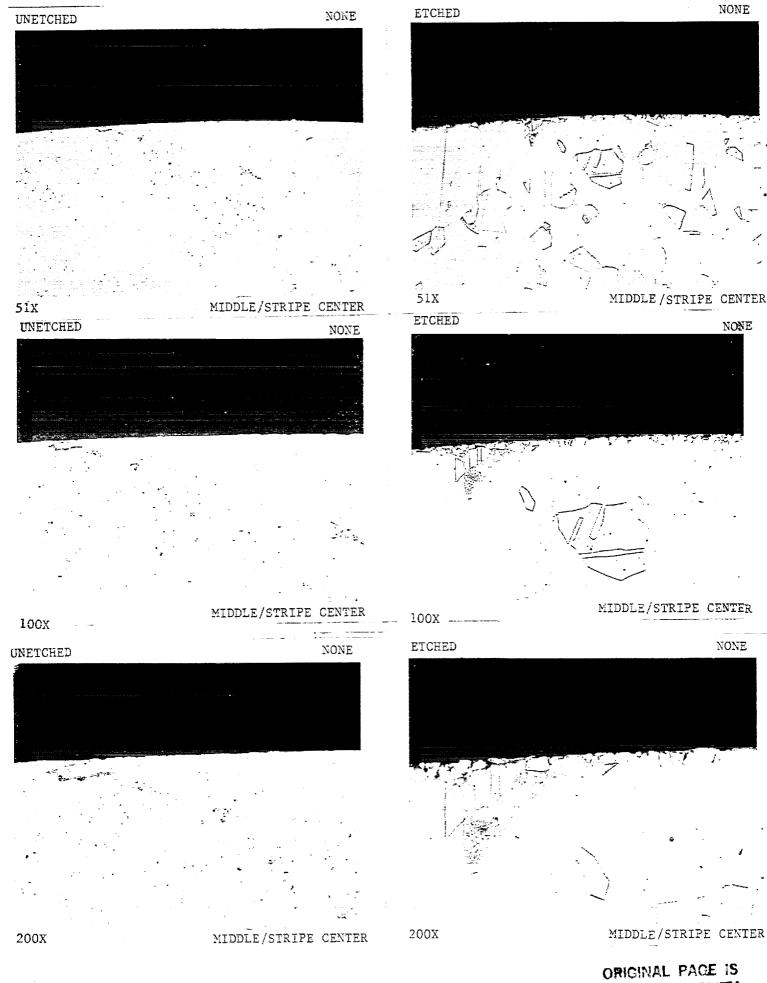
COATING = NONE OR NICKEL ALUMINIDE (N:AL)

MAGNIFICATION = 51, 100, OR 200 TIMES ACTUAL SIZE

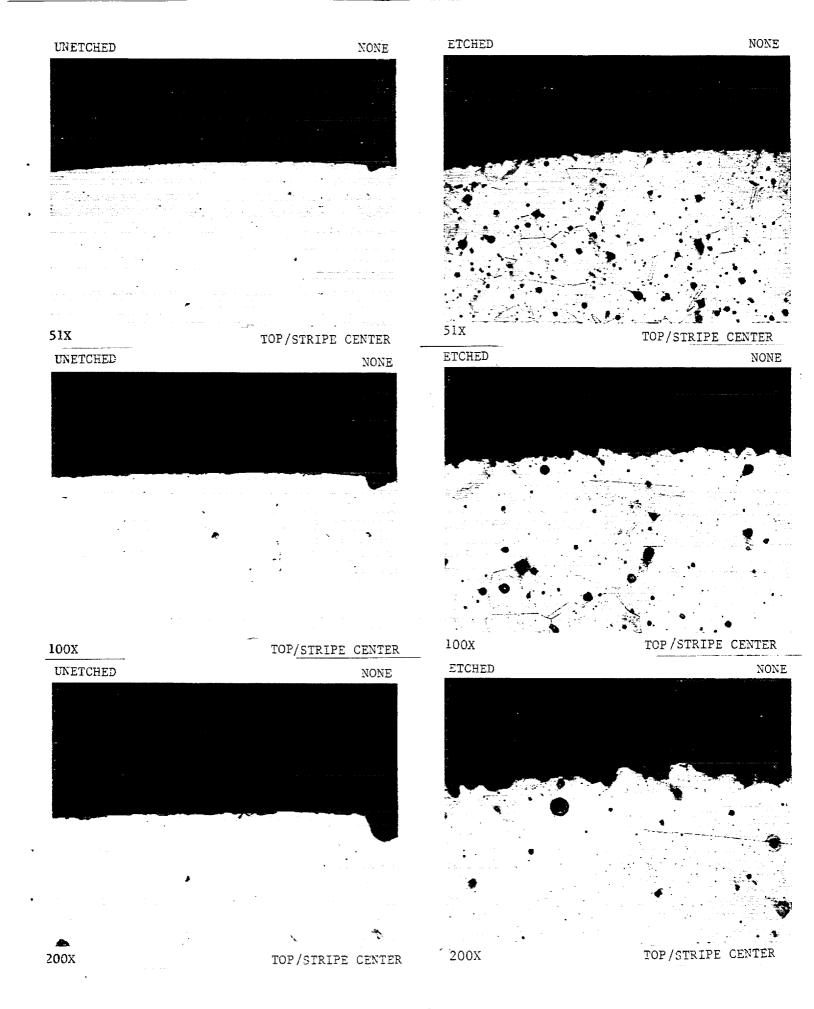
LOCATION = (TOP, MIDDLE, BOTTOM) /
(STRIPE CENTER, STRIPE EDGE, BETWEEN STRIPES)

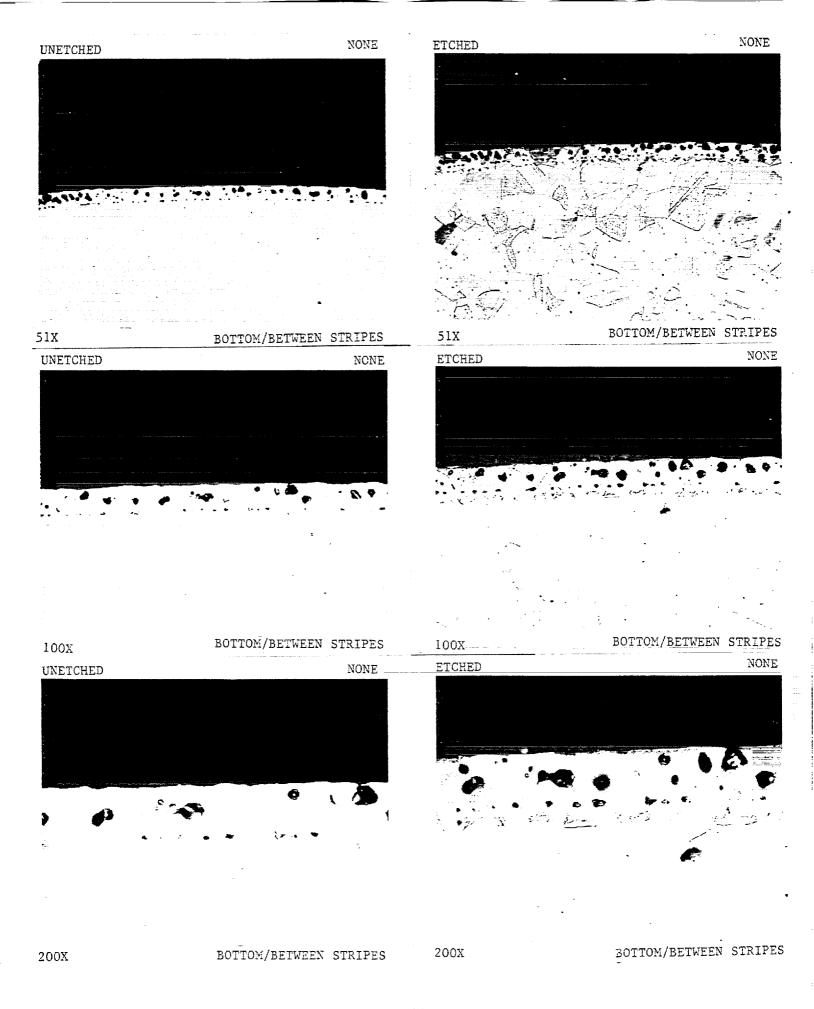
NOTE: REFER TO FIGURE 16 "KEY FOR AXIAL AND CIRCUMFERENTIAL PHOTOMICROGRAPH LOCATIONS" FOR INTERPRETATION OF SAMPLE LOCATIONS. THE BASE MATERIAL FOR ALL SAMPLES IS NICKEL 200.

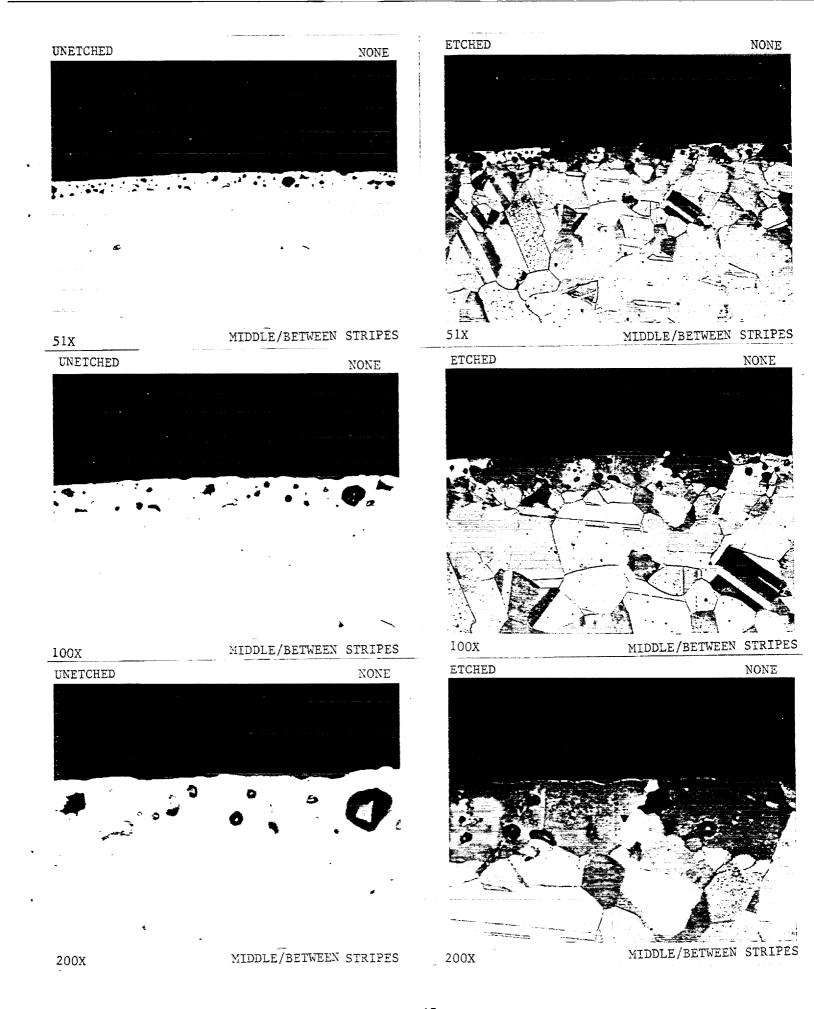


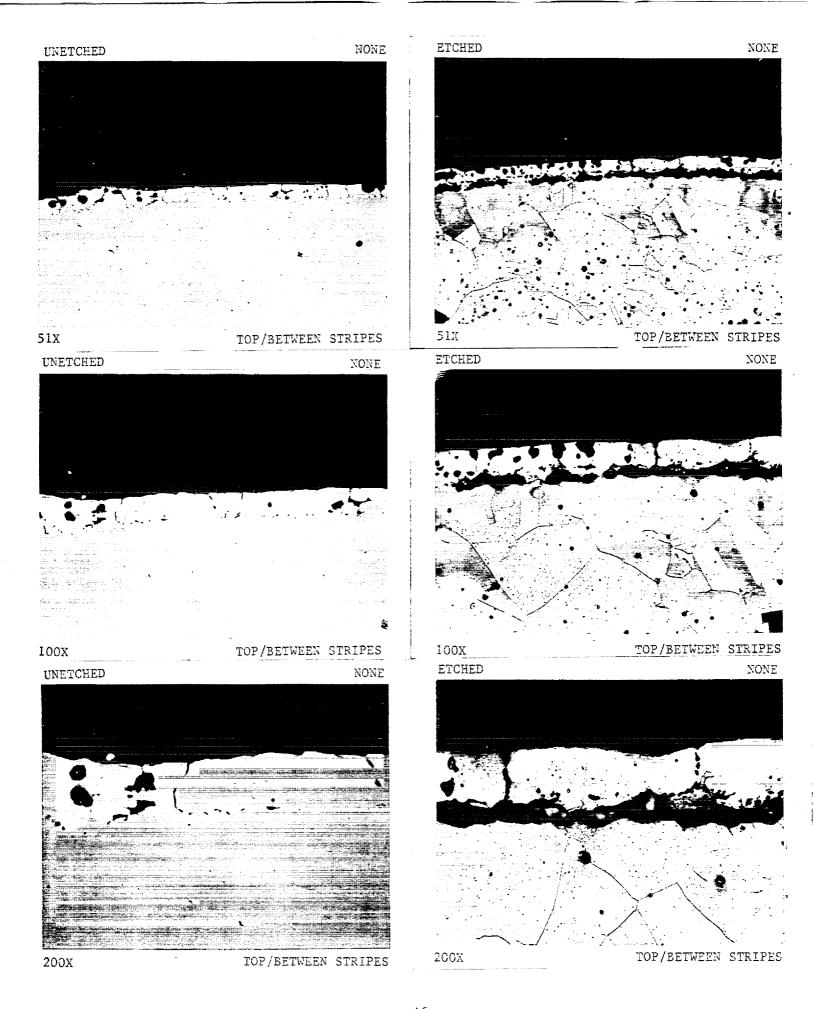


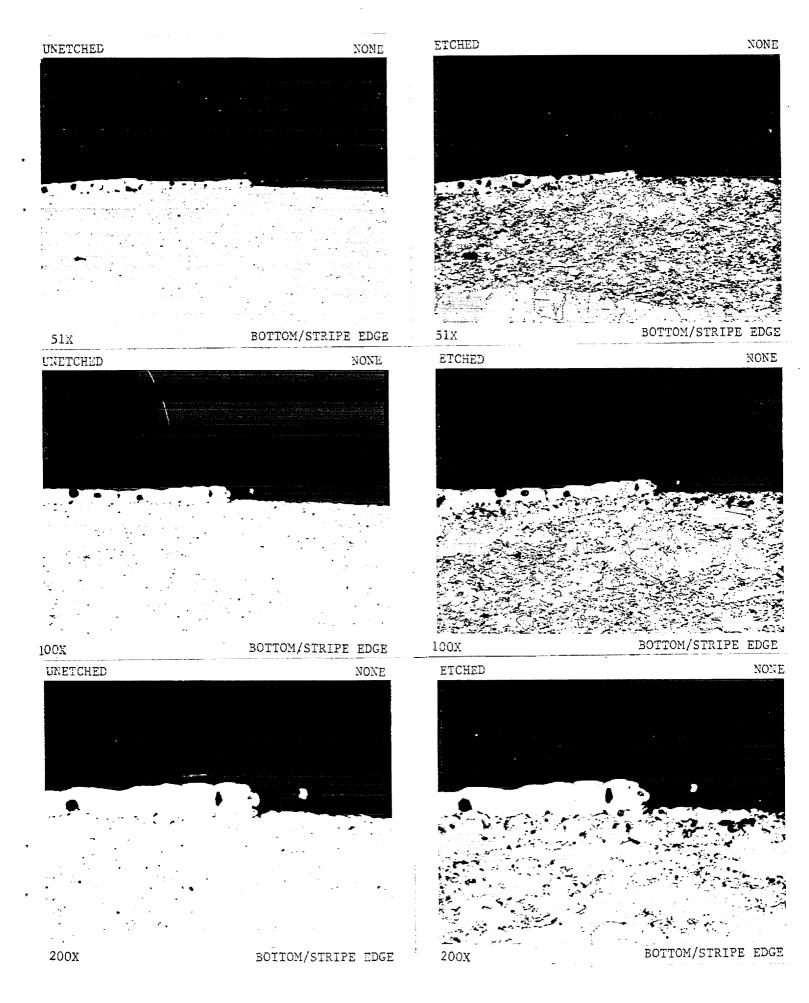
OF POOR QUALITY

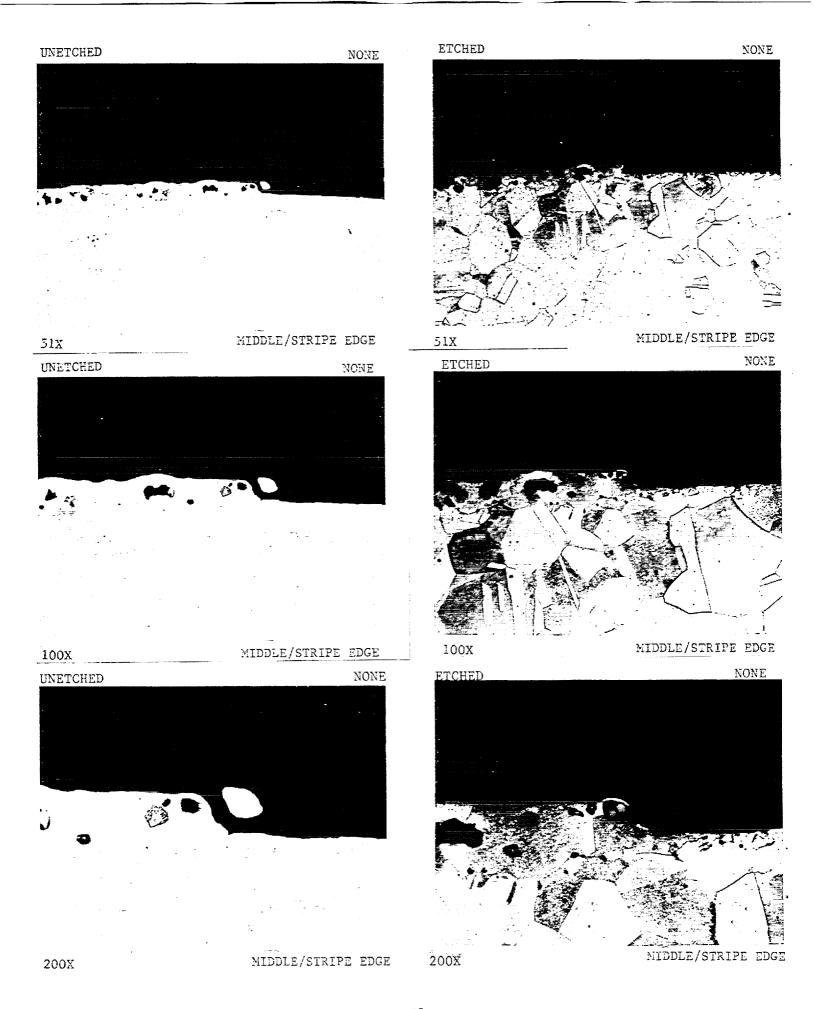


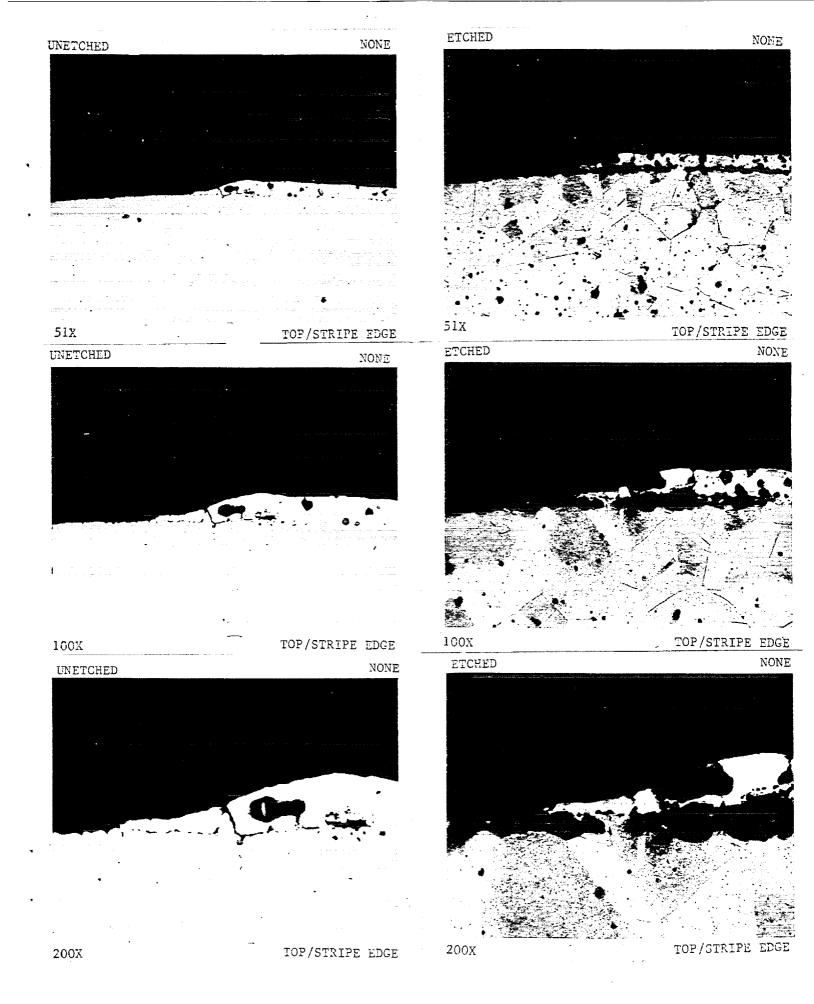


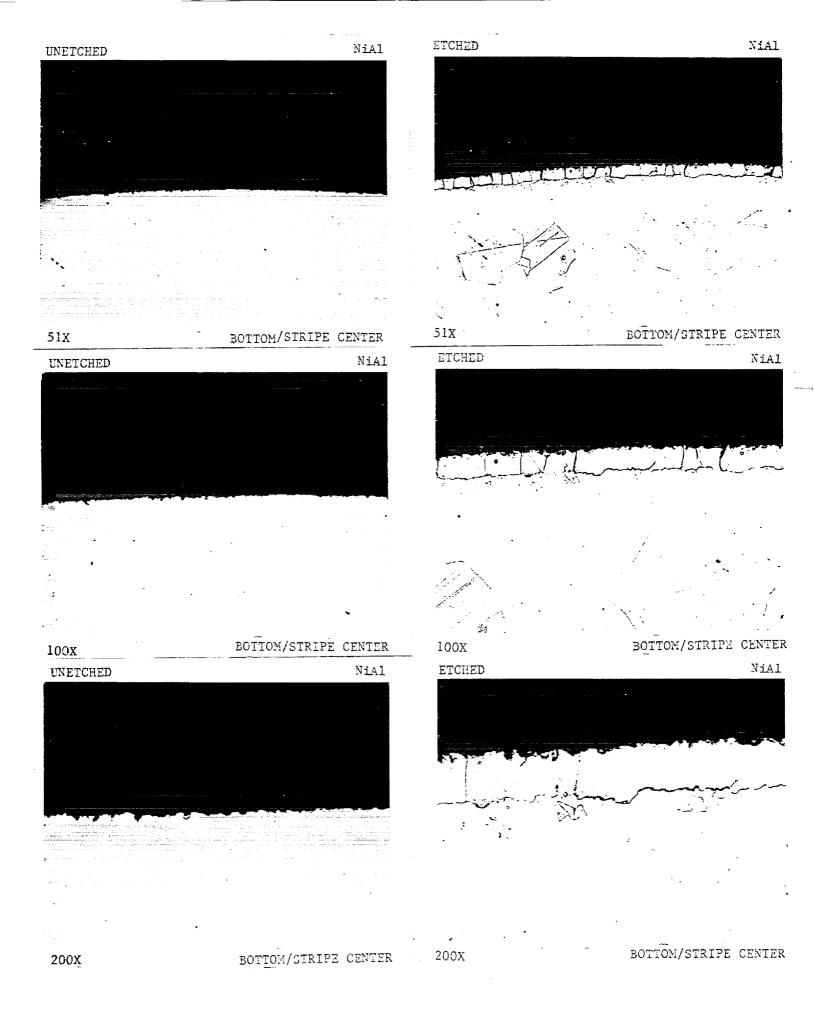


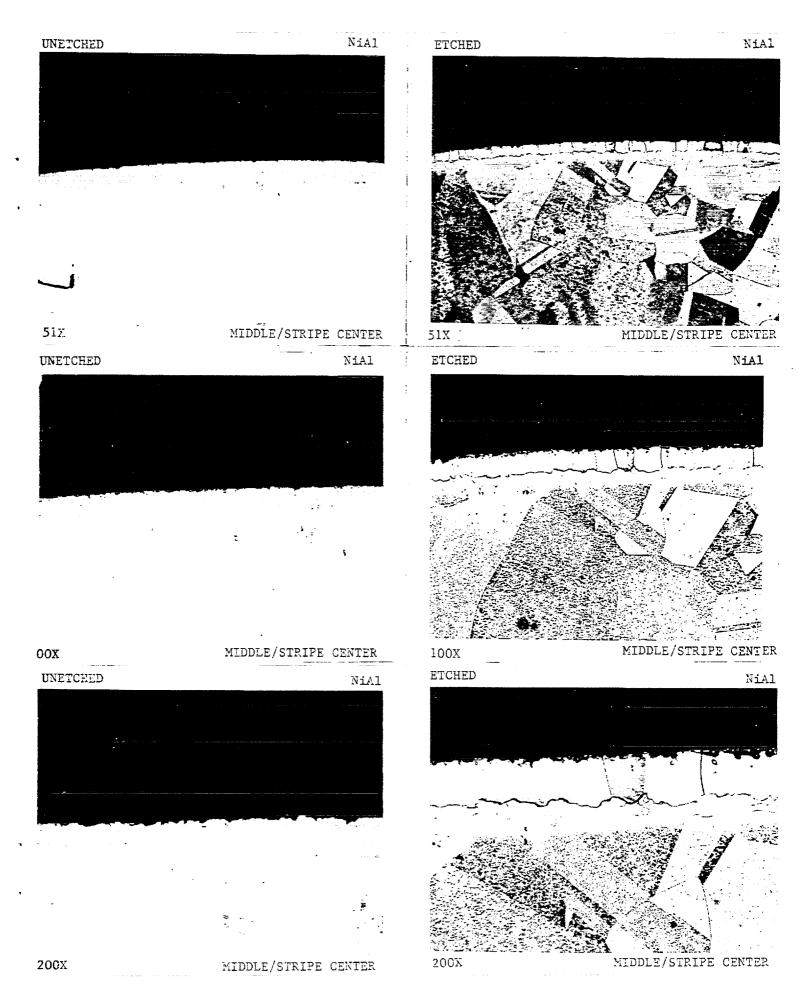


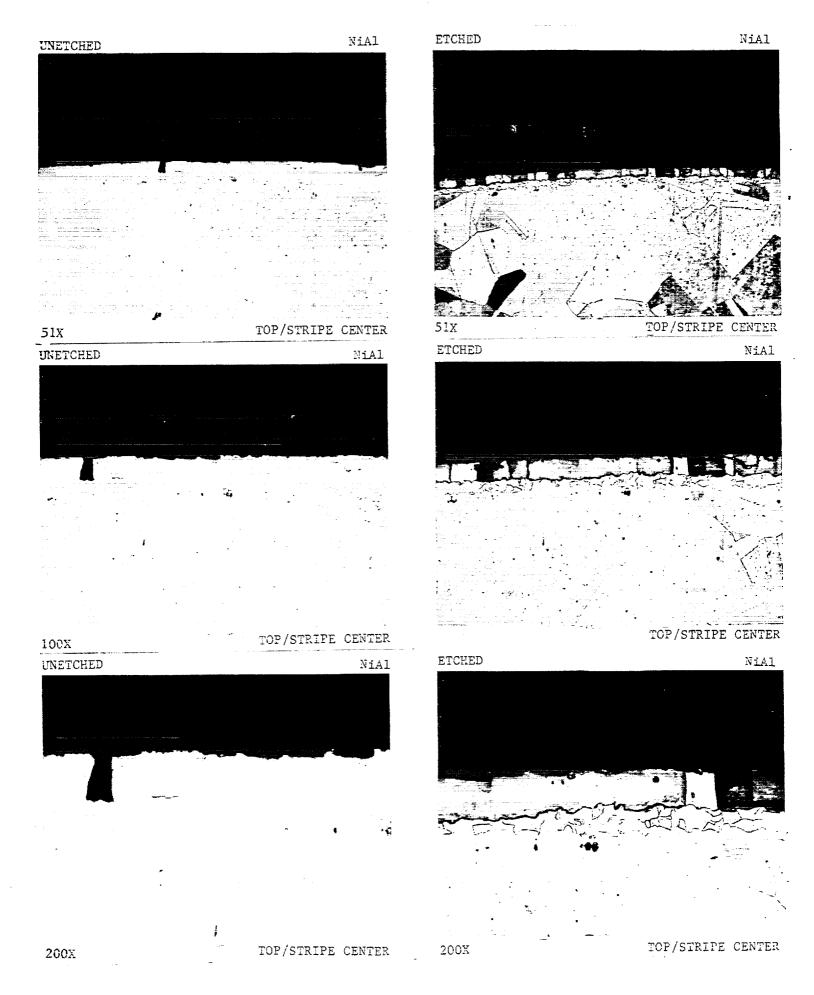


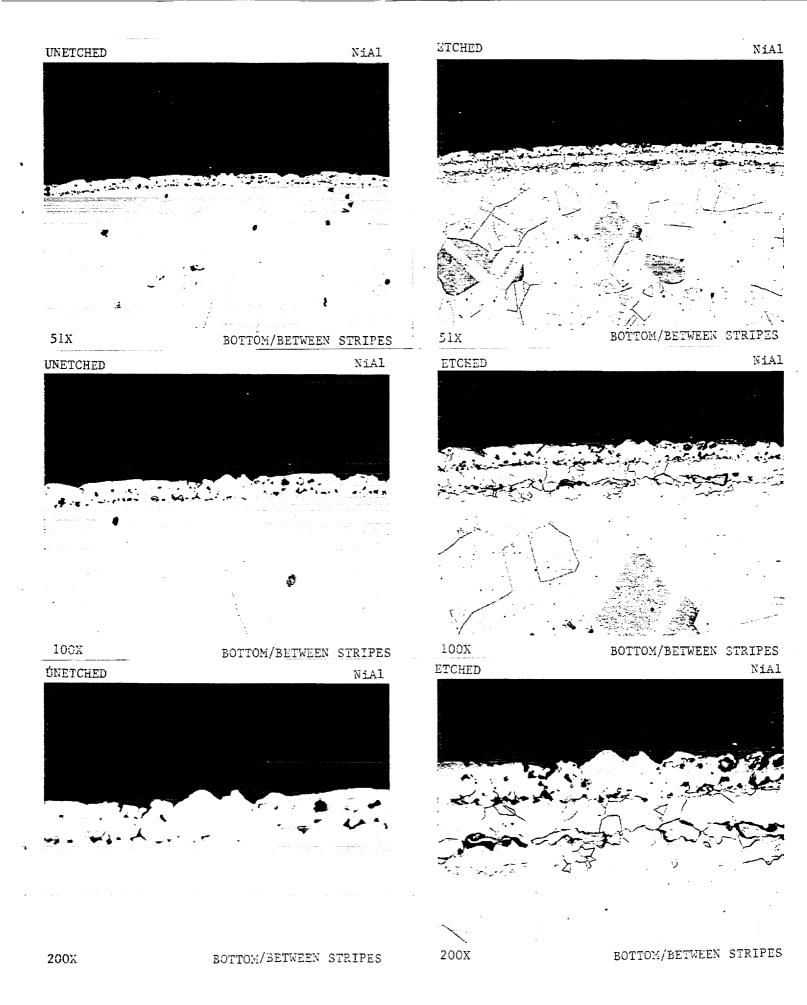


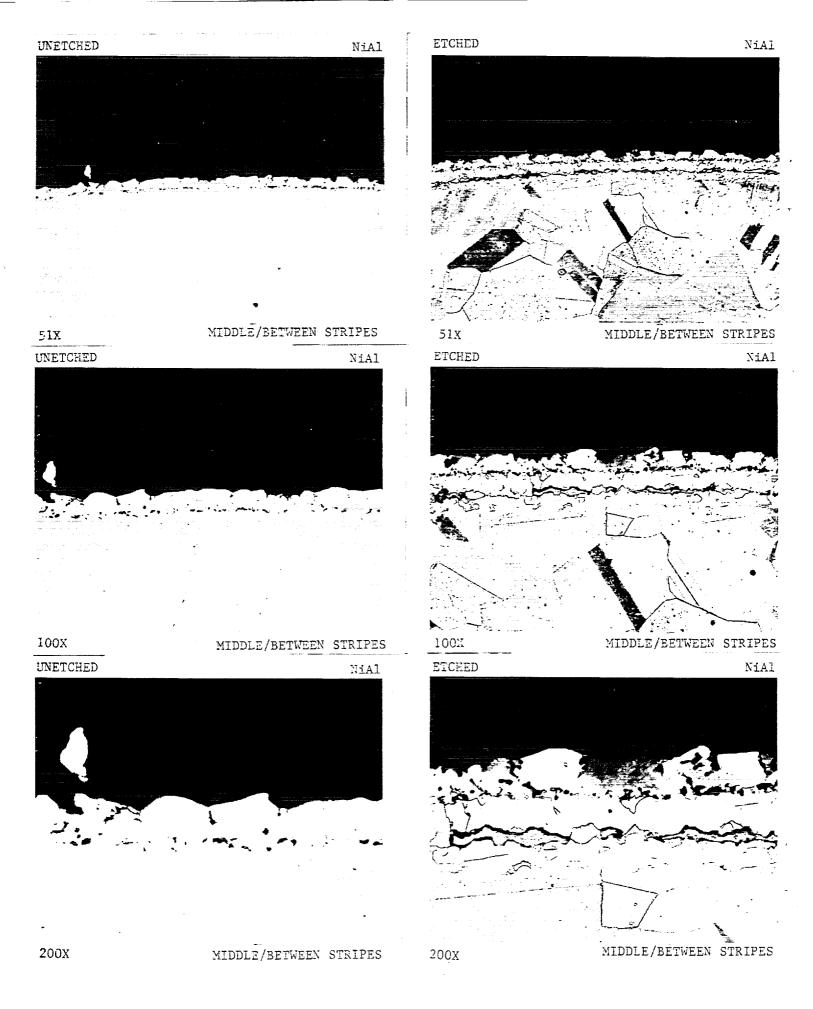


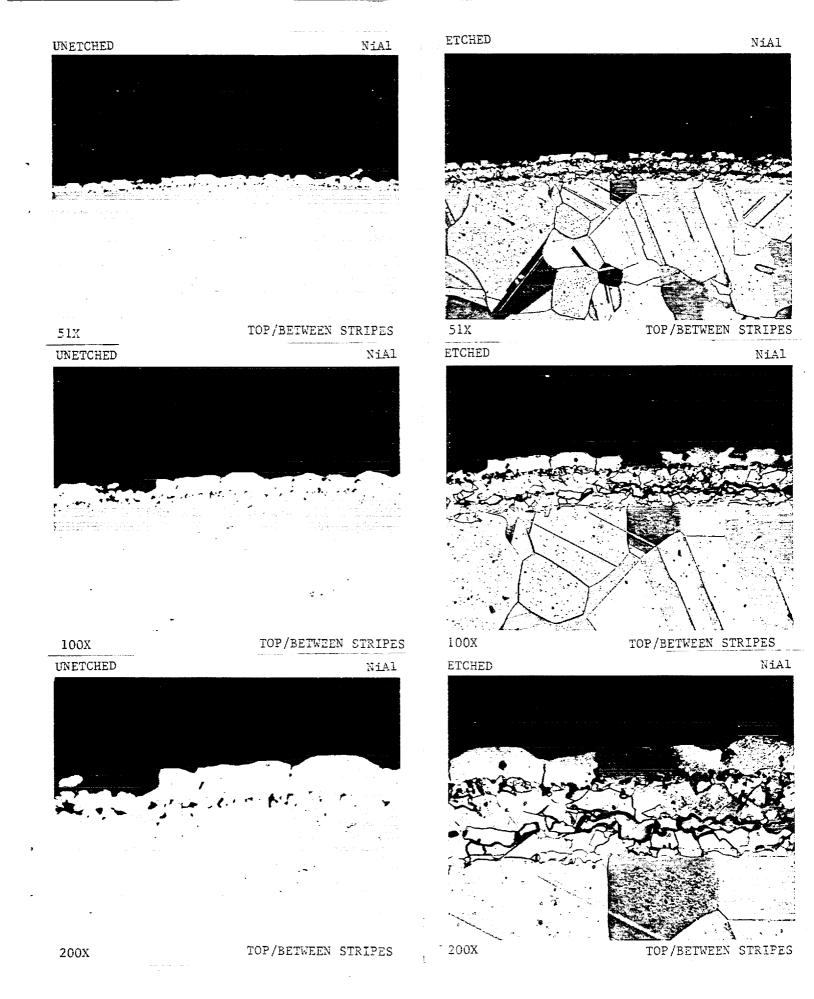


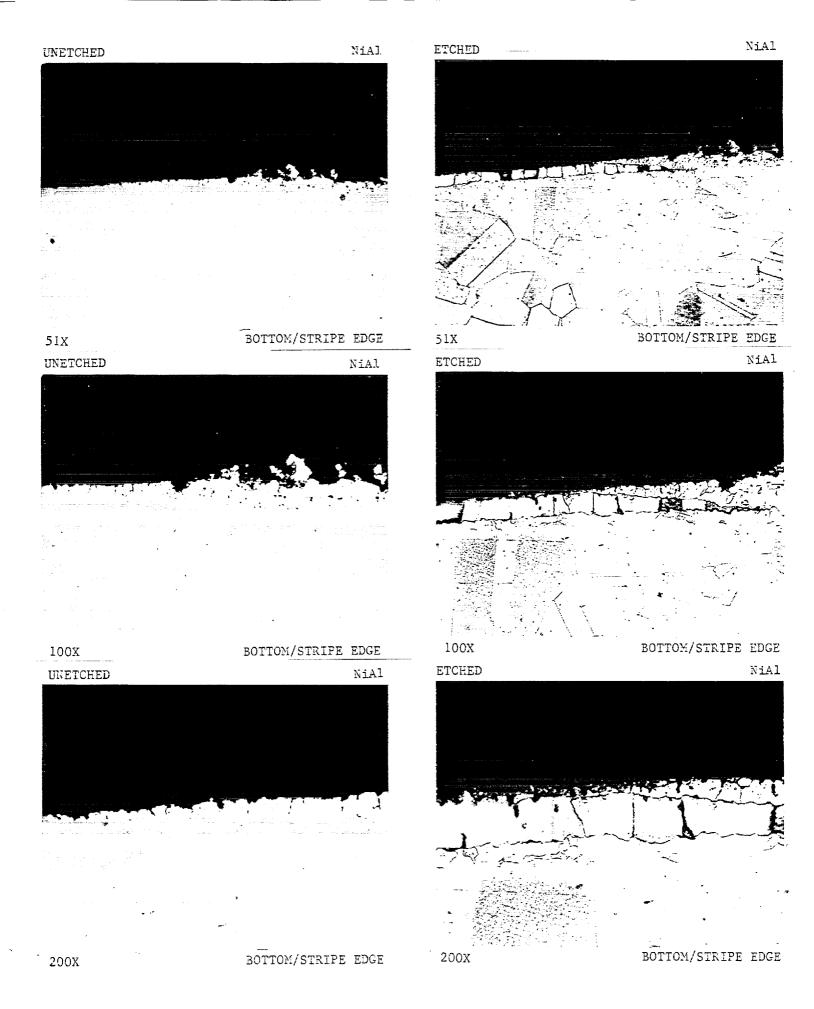


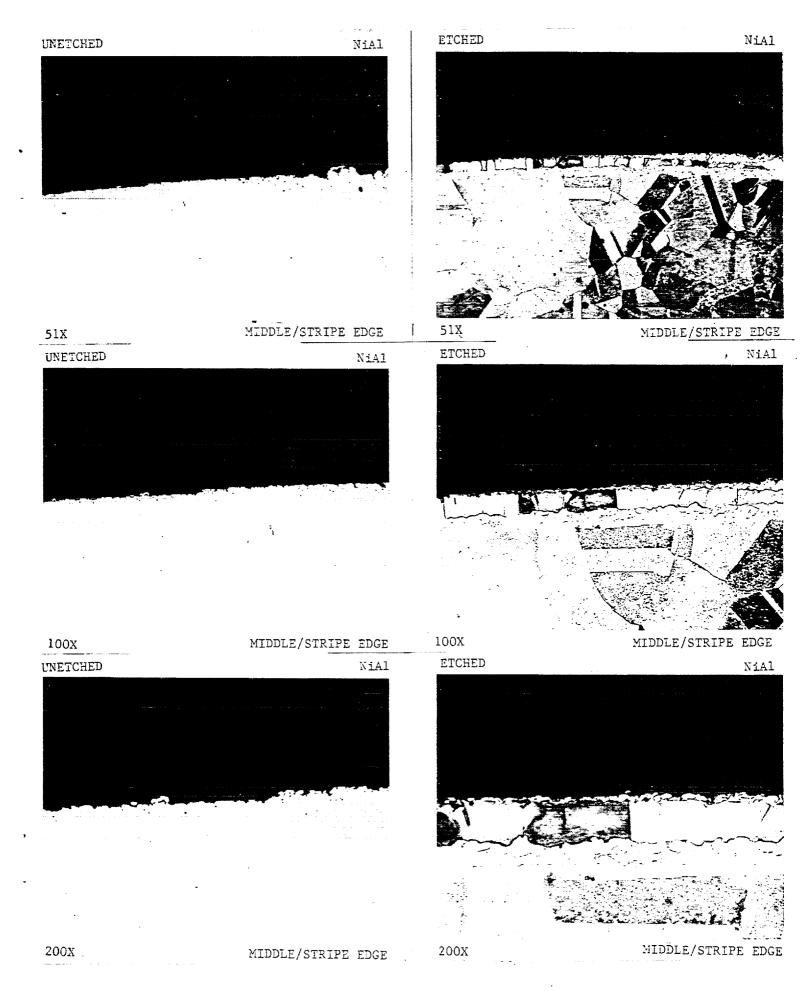


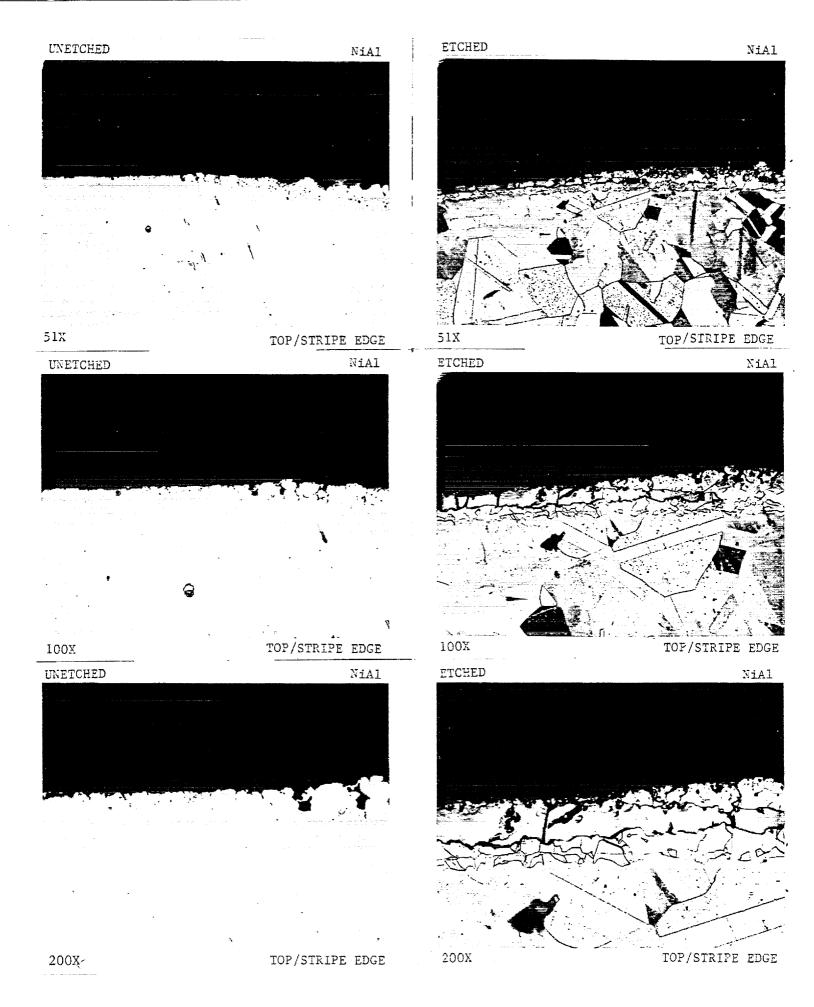












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13. ABSTRACT (Maximum 200 words)			
This report describes the work done by Administration Contract Number NAS: between January 1992 and July 1992. Sorbital and planetary missions. Dish Stirling engine electrical power general temperature and heat flux. Liquid meta uniform high temperature to the heat in potential life span. One issue that is rai heat input section by the liquid metal w based Stirling engine heater head mater aluminide as a coating and developed a and nickel aluminide coated Nickel 200 samples showed no visible sign of solu a resolution of better than 2.5 µm (.000 loop data, that nickel aluminide is still definitive conclusion whether coatings developed.	3-26325, "Insoluble Coatings for St Stirling heat engines are being devel- irling solar systems and nuclear reac- tion technology. The sources of them I heat pipe receivers are used as ther put section of the Stirling engine. The sed during the design phase of heat provided in the design phase of heat provided in the stirling engine. The soluble of heat pipe test velong the stirling engine in the own were tested for 1000 hours at 800 of the billity corrosion of either coated or used in the heat a leading candidate for solubility co	cirling Engine Heat Pipe Condenser Sur- oped for electrical power generation us ctor Stirling systems are two of the mos- mal energy used to drive the Stirling en- rmal transformers and isothermalizers to the use of a heat pipe receiver greatly en- pipe receivers is the potential solubility tiated a program to evaluate and demon- in sodium, potassium, and NaK. This pendid that can be used to test candidate to the condensation heat flux of 25 Walling and that complete the configuration of the configuration of the configuration protection, and that longer dura- times of the configuration of t	faces". The work was performed e on manned and unmanned earth t promising applications of the gine typically are non-uniform in the odeliver the thermal energy at a subances system efficiency and the corrosion of the Stirling engine strate coatings, applied to nickel program initiated a study of nickel materials and coatings. Nickel 200 cm ² . Subsequent analyses of the die, photomicrographs at 200X, has boarable to liquid metal pumped tion tests are required to reach a
	60		
Stirling engines; Heat pipes; Li	quid metals; Corrosion; Coat	tings	16. PRICE CODE A04
17 SECURITY OF ASSISTED TION 19	SECURITY OF ASSISTATION	40 SECURITY OF ASSISTATION	20 LIMITATION OF ARSTRACT

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